Corrosion

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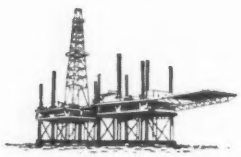

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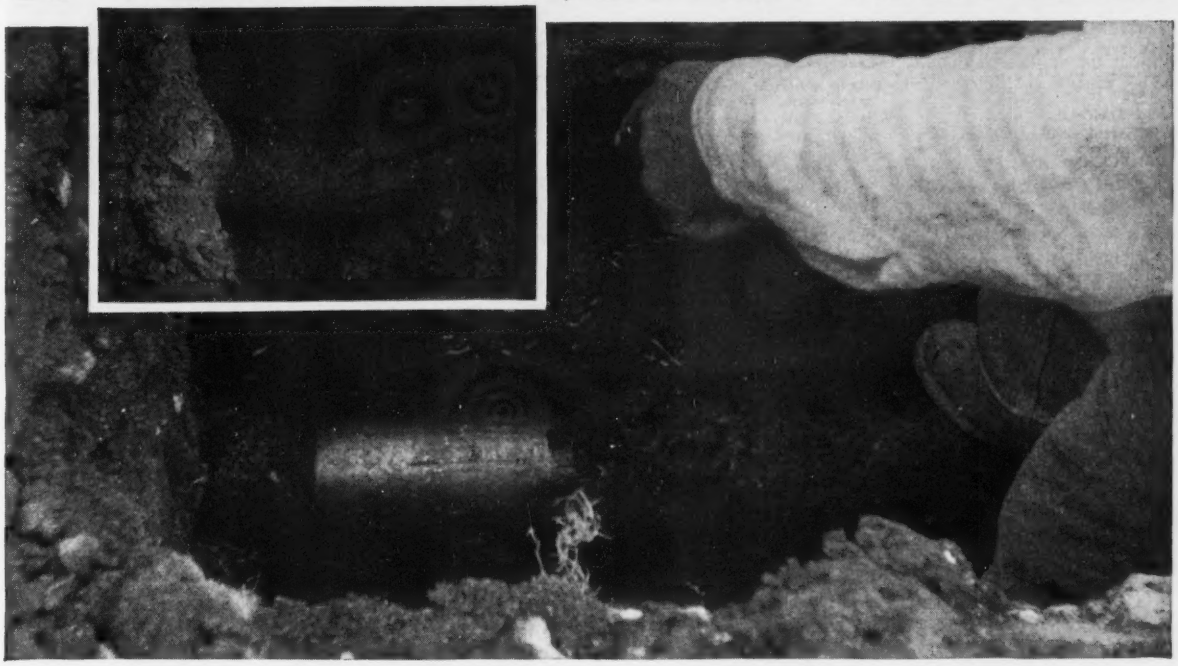

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T-3A-3 Materials Available for and Application of Corrosion Inhibitors

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T-3C Annual Losses Due to Corrosion

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T-3D-1 Electrical Holiday Inspection of Coatings

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T-3E-2 Corrosion in Railroad Hopper Cars

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T-3F Corrosion by High Purity Water

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T-3F-2 Inhibitors

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T-3F-3 Corrosion Products

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T-3F-6 Intercommittee Activities

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T-3F-7 Bibliography

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T-3G-1 Cathodic Protection of Hull Bottoms of Ships

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T-3G-2 Cathodic Protection of Heat Exchangers

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T-3G-3 Cathodic Protection of Process Equipment

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T-4H Tests and Surveys of Cable Sheaths

T-4J Cathodic Protection of Cable Sheaths

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T-5D-8 Methods and Criteria for Evaluating Plastics in Chemical Environment

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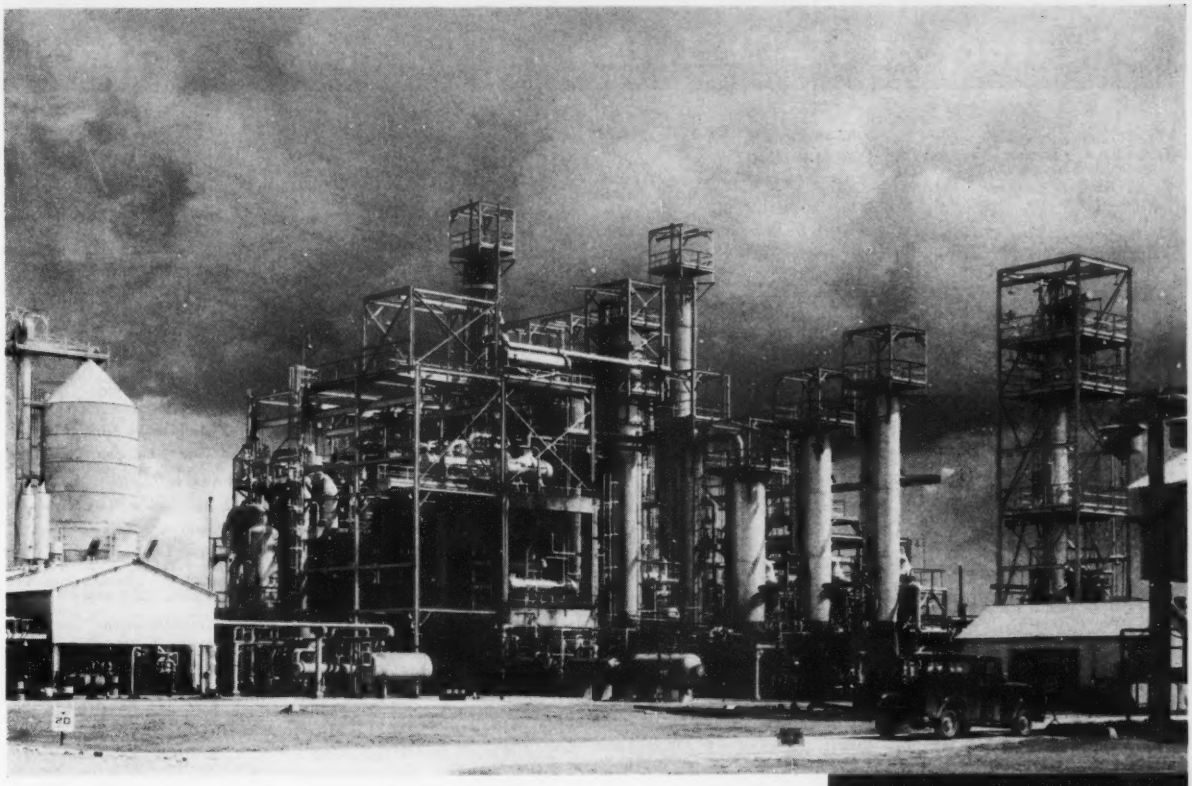

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T-6A-19 Asphalts

T-6A-20 Plastisols

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T-6A-21 Compile Reports for Binding Into One Volume

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T-6D-2 Standardization of Scope of Painting Specifications

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T-6H-1 Glass Coatings

T-6H-2 Porcelain Enamel Coatings

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T-6J-3 Inspection Techniques

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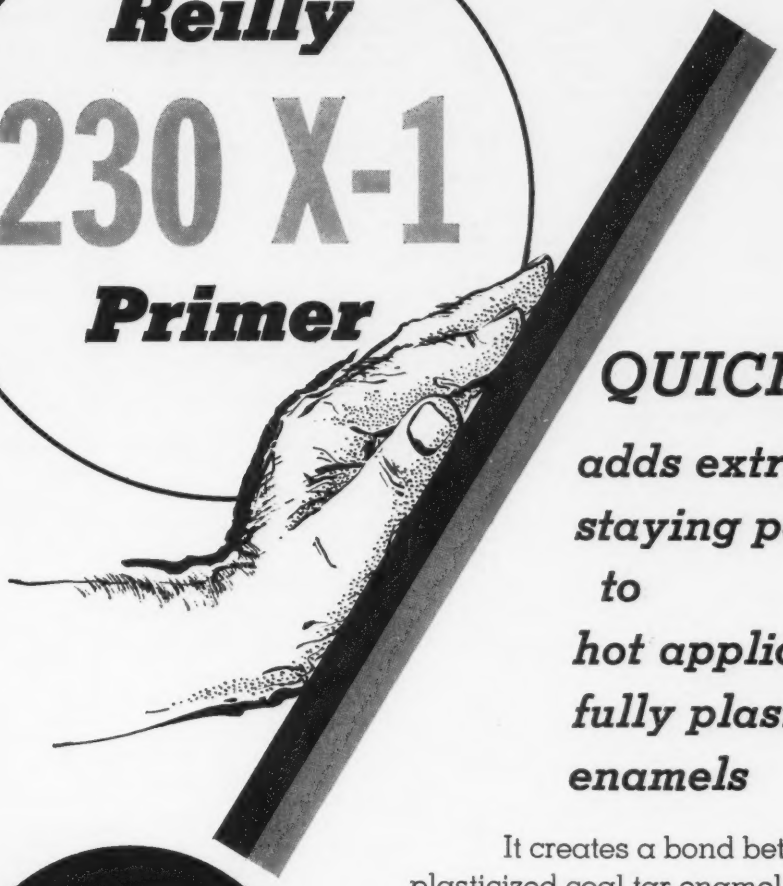

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Controlling Internal Corrosion of Tank Ships, by J. Franklin Koehler

New Instruments and Techniques for Ultrasonic Measurement of Tank Ship Corrosion Losses, by Dwight J. Evans.

Effects of Foreign Metals on Corrosion of Titanium in Boiling 2M Hydrochloric Acid, by Roger Buck III, Billy W. Sloope and Henry J. Leidheiser, Jr.

Instruments for Measurements in Underground Corrosion Work, by K. G. Compton

Equivalent Electrical Circuit Analogy of Structure-to-Soil Potentials, by Roy O. Dean

Service Experience With Lead-Silver Alloy Anodes in Cathodic Protection of Ships, by K. N. Barnard, G. L. Christie and D. G. Gage

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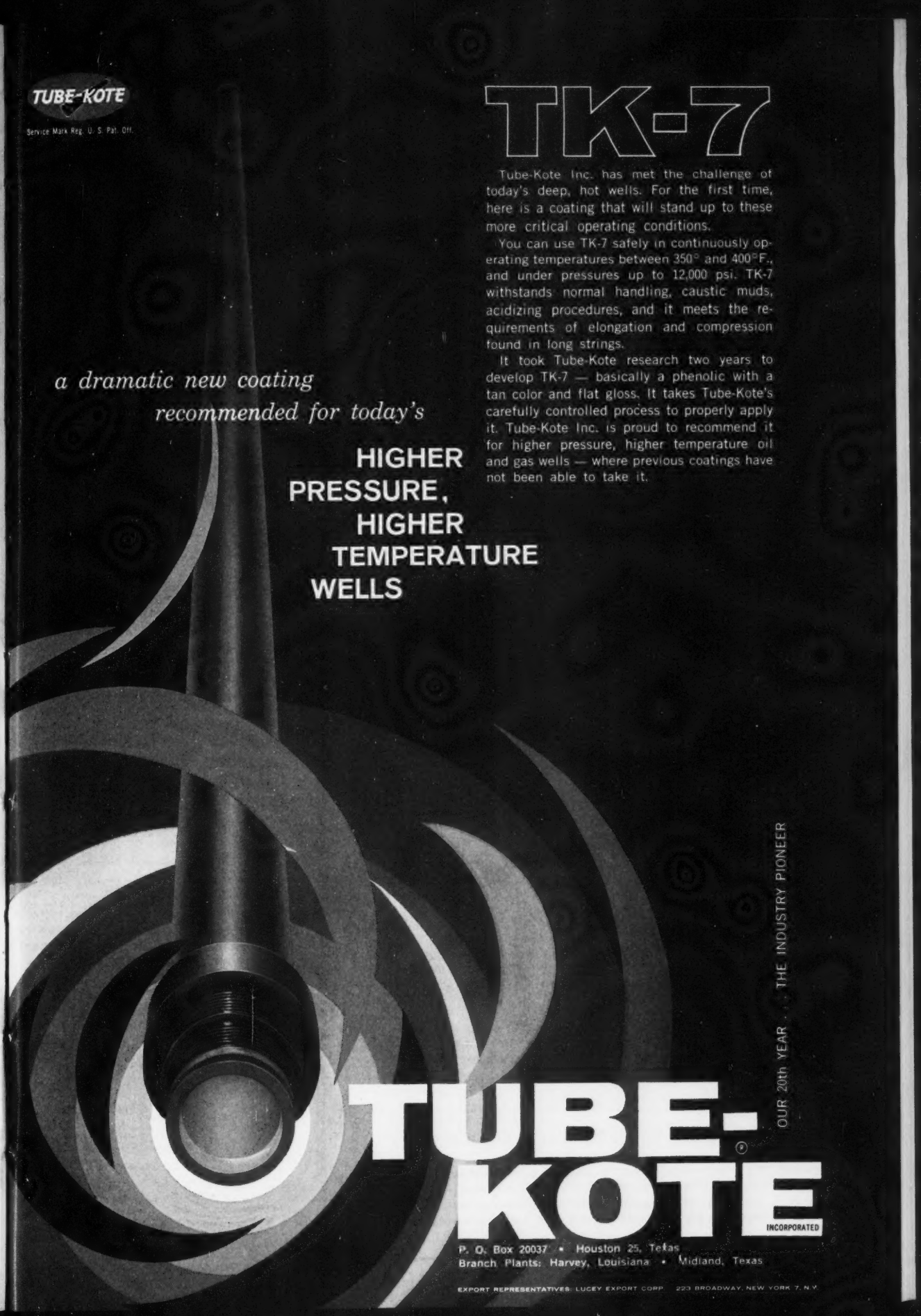

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Justification and Economics

Engineering Approach to a Paint Testing Program*

By D. A. BALLARD and V. B. VOLKENING

Abstract

A method of estimating the need for, size and potential savings to be expected from a paint testing program is given. This involves postulating a hypothetical chemical plant and evaluation of the economics, magnitude, savings to be expected, organization of the program, consideration of testing variables, design details, evaluation of results and other considerations. 34 references. 2.2.2

Ballard

Volkening

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Introduction

THE DECISION to establish a paint testing program in a company is dependent primarily upon the size of the maintenance painting expenditure. Usually the decision results when the expenditure appears excessive. At that time management must be willing to risk a sum equal to a fraction of this cost to investigate methods for reducing this expense.

Returns, in the form of dollars saved, are as much anticipated as if dollars were invested to manufacture a product from which a profit could be obtained. The size of the maintenance painting expenditure permits making an estimate of possible savings in the same manner that gross sales are estimated from the market potential for a product. Research cost must be only a fraction of the savings just as the cost of sales must be a fraction of the gross sales.

Hypothetical Chemical Plant

In order to better understand the problem visualize a hypothetical chemical plant (HCP). The HCP has a maintenance painting bill of $500,000 annually. It is decided that this expenditure is excessive and must be reduced. Starting with only the size of the maintenance painting expenditure much can be theorized further about the HCP until a fairly comprehensive picture is created. This picture permits the corrosion engineer and his management to compare their plant with the HCP. They know their present painting cost and this cost is the basic information needed to calculate the size of the research program required to produce maximum savings. The HCP is obtained from figures based on national averages which admittedly allow considerable variation, but accepting these averages aids in the development of a systematic program.

From published data, annual painting expenses of chemical plants is of the order of 0.4 to 0.5 percent of the capital invested.[1] This places HCP's capital investment in the order of $100,000,000 to $125,000,000. By using statistics available from the Conference Board Business Record[2] (which indicates the capital invested per employee in the chemical industry was $25,479 in 1954), the plant is estimated to have approximately 3900 to 4900 employees.

Carrying the analysis a step further assume that the applied cost per gallon of paint is $25.[3] This means 20,000 gallons of paint are purchased annually to maintain this plant, enough paint to cover 1,000,000 square feet of surface with six mils, assuming an average coverage of 300 mil-square feet per gallon. A million square feet of surface could include forty 40-foot x 40-foot tanks, 50 miles of pipe ranging from 2 to 12 inches in diameter and 500,000 square feet of vessels and structures. If it is assumed further that this paint system has an average life of five years, a total paintable surface of 5,000,000 square feet for the entire plant can be calculated.

At an average cost of $6 the 20,000 gallons cost $120,000, or 24 percent[4] of the total paint maintenance cost. Adams[3] says that the consensus of several investigators indicates that surface preparation accounts for about 50 percent of the total cost. This would allow 26 percent or $130,000 for the paint application.

TABLE 1—Hypothetical Chemical Plant

Capital investment	$100,000,000—$125,000,000
Number of employees	3900—4900
Surface area painted annually, sq. ft.	1,000,000
Annual maintenance painting expense	
Surface preparation 50%	$250,000
Application 26%	$130,000
Paint 24%	$120,000
Total 100%	$500,000
Volume of paint consumed, gallons	20,000
Applied cost per square foot	50¢
Service cost per square foot/year	10¢

Estimating Maintenance Research Program

Now to get back to the information upon which management must base its decision. It has been established that it is desirable to reduce the $500,000 annually paint maintenance bill. Tator[5]

TABLE 2—Size of Annual Maintenance Research Program With Indicated Savings

Maintenance painting expense	$500,000
Indicated possible reduction	20—50%
Total savings estimated	$100,000—$250,000
Maintenance research budget	$10,000—$25,000
Number of paint test panels	400—1000
Reduced maintenance painting expense resulting from research program	$275,000—$390,000
Hypothetical cost per Square Foot	27.5—39.0
New service cost per square foot per year	5.5—7.8

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Figure 1—A paint test panel.

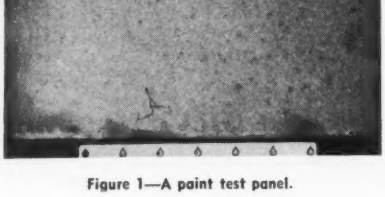

Figure 2—Change in over-all average grades with time.

TABLE 3—Variables of a Typical Paint Test

Variable	Level	Type
Vehicle	6	Qualitative
Surface preparation	2	Qualitative
Film thickness	3	Quantitative
Atmosphere	2	Qualitative

has indicated that 20 to 50 percent savings can be expected from a paint testing program. Assuming a median value of 35 percent, the amount of money to be set aside for a paint maintenance research program can be estimated. And if return on research dollars is to be about $10 for every dollar invested, approximately $17,500 per year can be spent on this program.

The $500,000 being spent to coat 1,000,000 square feet of surface, produces an applied cost of 50 cents per square foot. With a service life of 5 years, the cost per square foot per year subsequently should be reduced from 10 cents to 6.5 cents. These data are given in Table 2.

Now a maintenance research program must be developed with the money available. A paint panel testing program, the most economical way to obtain the required data, is analagous to the test tube or laboratory bench scale of chemical research. The number of panels needed to give the desired information can be estimated.

Cost of application and exposure may be as much as $10-$15 per panel and by the time the panel has been exposed, evaluated and results reported, the cost may be in the order of $20-$30 per panel. If a figure of $25 per panel, is taken, this will give an indication of the number of panels which may be exposed annually. With an annual budget of $17,500 and an estimated cost of $25 per panel exposed, it follows that 700 panels which may be exposed. To get the most out of the panels exposed, it is highly desirable that each exposure series be designed to obtain the maximum information, an objective which will help reduce the number of panels, increase the amount of information and reduce the maintenance research cost. For this reason, statistical design finds an important place in paint panel testing.[6]

Design of the Panel Test Program

Determine the Purpose of the Test

The first thing to be done is to establish definitely the variables that will be under consideration in the tests. Many paint test programs produce mediocre results because of improper choice of variables to be investigated. Major groups of independent variables that can be studied are Paint formulation, Substrate, Surface Preparation, Paint Application, Atmosphere.

Of the large number of formulation variables,[7,8,9,10] only two will be mentioned, the effect of vehicle and the effect of pigments, both corrosion inhibitive and color. The paint manufacturer has complete control of the paint formulation variables; therefore, good liaison between the corrosion engineer doing the paint testing and the manufacturer is essential. The corrosion engineer should be sure that the paint he is testing is the same as that the manufacturer can and will produce.

The effect of substrate [11,12] can be studied. In addition to carbon steel, paint may be applied over galvanized steel, corrosion resistant alloys, or nonmetallic materials.

Surface preparation[13,14] is an important variable. Mechanical and chemical surface preparation must both be considered. An important variable in all painting programs, and one that may be most significant, is the application. A familiar example is film thickness.[15] The best way to control the painter, the most important variable in paint application, is to have him properly trained, informed and supervised.[16] The method of application also is important. This can be by brush, roller, or spray[17] (conventional, hot or airless). The physical properties of the paint during application (e.g., viscosity) are important. Weather conditions[18] during the application and between coats must be considered.

The effect of the atmosphere during exposure also varies. The atmosphere may be very corrosive, or it may be mild.

Design of the Experiment

After a clear definition of the independent variables to be tested has been established the next step is to design the experiment. Most of the independent variables under consideration in paint testing such as vehicles, pigments, substrates, or methods of application, are QUALITATIVE. The person doing paint testing seldom has the advantage of such QUANTITATIVE variables as temperature, pressure or concentration. The most useful experimental design for paint testing is a factorial design;* i.e., a panel for all possible combinations of variables in the test.

Consider an example. The corrosion engineer at the HCP has six different vehicle systems available. His plant has an acid atmosphere and an alkaline atmosphere. His maintenance painting is done over both sandblasted steel and hand cleaned steel.

Film thickness is another variable. Table 3 shows the variables and levels involved.

Without replication he must prepare 6x2x3x2 = 72 panels (i.e., 6 vehicles, 2 surface preparations, 3 film thicknesses, 2 atmospheres) to have a factorial design. Each year he could prepare ten different experiments of this size within the limits of the 700-panel budget.

Other experimental designs can be used. In the event of limitations such as the amount of paint available, number of panels, rack space, or economics, one of these other experimental designs can be used. The bibliography lists several important texts on the subject of experimental design.[19,20,21,22]

It is important to control as closely as possible the variables under study. All variables, both independent and dependent, are subject to experimental ERROR. For example, the HCP corrosion engineer selected 2, 4 and 6 mils as film thicknesses in his paint tests as levels of an independent variable. The differences between the designed and applied film thickness contributes to experimental error. Differences in panel grades for the dependent variables contribute to experimental error. It is important to know the size of the error; i.e., get an estimate of the error.[6] A good estimate of the error is essential for good results from the paint test. Replication of panels can assist in obtaining a better estimate of the experimental error.

In any paint test of practical dimensions there are many variables **not** a part of the experiment design. Some of these other variables may be very critical if not controlled closely. Controlling these variables is called controlling EXTERNAL BIAS. A list of some of the methods of controlling bias in a paint test are given. It is not inclusive.

1. Use individual panels instead of the paint stripe method. (See Figure 1)
2. Cut the panels from sheet steel and make a random selection of each panel before it is prepared.
3. Protect the backs of the panels.
4. Use one painter to prepare all panels.
5. Be sure that the panels are all painted in the same area to prevent contamination of part of the panels and not the rest.
6. Use the same method of application throughout the test.
7. Arrange to have similar drying times between coats.

* O. L. Davies. "The Design and Analysis of Industrial Experiments," pp. 296-303, Oliver and Boyd, London, 1954.

8. Prepare the panels to give an outward appearance (for example, color) as similar as possible.
9. The panels should be randomized before they are attached to the racks.
10. The panels are best attached to the racks by ceramic insulators to prevent electrochemical contact between panels or the rack itself.
11. The panels should be exposed in the same general plant area.

The interval of evaluation should be a part of the experimental design and related to the expected life of the panel series. The intervals should be equal or multiples of equal intervals to make evaluation of results simpler. Figure 2 shows what can happen if the panels are evaluated too soon (for such a dependent variable as rusting). The natural desire of the grader to have a spread of data may cause the average grades at the first inspection to be too low.

Grading Standards

Dependent variables in paint evaluation[23] are rusting, loss of adhesion, blistering, pitting; checking, alligatoring, cracking; flaking, scaling, peeling; lifting, wrinkling, chalking.

The variables to be used are decided upon before the test begins. Obtain standards for each dependent variable that is to be graded. The American Society for Testing Materials and the Federation of Paint and Varnish Production Clubs provide such standards.[24] The photographs and grades for each type of failure are excellent.

It may be advisable to weight the variables. For example, assume the dependent variables to be graded are: general appearance, checking, blistering and rusting. If the value for rusting were doubled the total value of rusting would be 40 percent of the final grade rather than only 25 percent.

Observer error generally is high.[25] For that reason, it is suggested that at least three observers grade the paint panels at each inspection. The panels should be graded on the same day, preferably at the same time of day (if the grading is done outdoors). A grading caucus before the inspection begins is helpful. The graders should reach reasonable agreement between the standards and the condition of the panels. All observers should record a grade from 0 to 10 (with 10 being perfect) for each dependent variable for each panel during an inspection.

A good color photograph of all panels should be made at the time of each inspection. This does not mean that a single photograph is necessary for each panel. A photograph can be taken of the whole rack containing from 10 to 100 panels so long as the detail desired is adequate. Figure 3 shows an example.

Evaluation of Results

After the grading is completed, data are available from which selection of materials and methods can be made with defined confidence. The method of statistical evaluation of these data is something that was decided upon when the experiment was designed.[16,20,21,26,27]

The data can be evaluated, using a desk calculator, but the amount of data compiled and the length of the calculations can very quickly make this method impractical. When this situation arises, the logical course to follow is to use punched cards and electronic calculating equipment. In most metropolitan areas there are service agencies that will analyze data for the investigator at a nominal charge.

The total score for each panel for each observer at each inspection should be calculated (100 as perfect). After these scores have been determined the data can be partially evaluated by simply arranging the scores in an order of merit. When several variables were weighed this becomes too difficult to be practical.

The most powerful analytical method is the ANALYSIS OF VARIANCE.[28] Analysis of variance is extremely important because of the large amount of information that comes from the calculations. The most important is that it measures the EFFECT of each variable in the test. That should have been the main reason for doing the test in the first place. From this measure of the effect of each variable, the investigator is able to tell not only that the effect is SIGNIFICANT (or that it is not) but get an idea of **how** significant. To have good significance, however, the test should have been designed in such a manner as to give an adequate estimate of the error.

A discussion of a recently completed paint test will illustrate the value of the analysis of variance. The experiment was designed to determine the concentration of an inhibitive pigment (red lead) necessary for use in vehicles of different steel wetting ability. The variables and levels are listed in Table 4.

Without duplication, a factorial design for this experiment required 4x4x2x2 = 64 panels. Table 5 given the analysis of variance for this experiment after 36 months' exposure.

Because the analysis of variance (Table 5) said all variables were significant except surface preparation, therefore, there are significant differences between some levels of the other three variables. (However, which levels these are is unknown.)

With only two levels, Top coat B is significantly better than Top coat A.

Calculations can be made[29] that show that only the difference between the 0 lb/gal and 8 lb/gal red lead concentrations is not significant (Table 6).

It is clear now that under conditions of the test several conclusions can be drawn with likelihood of being wrong only one time out of 20. They are: 1. Top coat B is better. 2. There is no difference among results achieved by the two methods of surface preparation. 3. The 4 lb/gal red lead concentration is best. 4. The vehicle with steel wetability of 7 is best.

Pitting

Pitting may be used as a quantitative dependent variable in evaluation of paints in an aggressive atmosphere. When nodular rusting (blistering) appears, the area immediately beneath the blister is a pit in the metal. The depth of a pit can be measured to give a quantitative value. Where pitting is considered of prime importance, this could be the only dependent variable studied. At the conclusion of the test all of the coating and rust is removed by alkali and acid pickling. After the final rinse some method should be used to prevent light rust from reforming if pit depths are not measured within a reasonable time.

The following method is suggested to evaluate pitting: Each panel is divided

TABLE 4—Variables of an Inhibitive Pigment and Vehicle Paint Test

Variable	Level	Type
Red lead concentration	0, 4, 8, 12 lb/gal	Quantitative
Vehicle	2, 4, 7, 10* wettability	Semi-quantitative
Surface preparation	Sandblasted Phosphoric acid pickled	Qualitative
Top coat	Top coat A Top coat B	Qualitative

* An arbitrary rating of **steel wetting ability** with 10 being best.

TABLE 6—Average Grades and Significant Differences for Each Level of the Variables in a Paint Test

Variables	Main Effect of Each Level	Average Grade (Average for all panels = 73.3)
Topcoats	Topcoat B Topcoat A	79.9 66.6
Surface preparation Methods	Sandblasted H_3PO_4 pickled	73.3 73.2
Red lead concentration	4 lb/gal 0 lb/gal 8 lb/gal 12 lb/gal	78.8 73.3 71.7 69.4
Vehicles	7 wettability 4 10 2	86.2 74.6 71.8 61.6

Note: Any two means bracketed are not significantly different.

TABLE 5—Analysis of Variance for Inhibitive Pigment/Vehicle Experiment

Source	Sum of Squares	Degrees of Freedom	Mean Square	F-Ratios	Significance
Top Coats	19,682	1	19,682	>3.86	Highly Significant
Surface Preparation	19	1	19	<3.86	Not Significant
Red Lead Concentrations	5,425	3	1,808	>2.62	Highly Significant
Vehicles	31,732	3	10,577	>2.62	Highly Significant
Error	22,638	393	58	....	.....

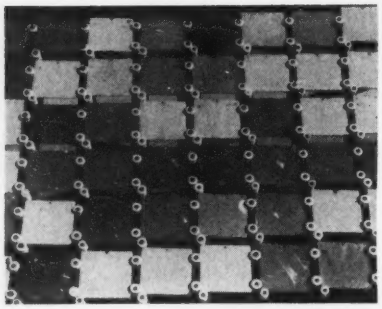

Figure 3—A rack of paint test panels.

into at least ten equal areas. The deepest pit in each of these small areas is measured using a pit depth gauge. The best evaluation of these data is an analysis of variance of the independent variables (e.g., concentration of inhibitive pigment) using the average pit depth as the dependent variable.

Some of the factors that apparently affect under-film pitting are: 1. Atmosphere. 2. Type of top coat. 3. Film thickness. 4. Inhibitive primer pigment concentration. (See Figure 4) Note the difference in pitting under the bare primers and under those with top coats. Compare the total destruction which occurred under the bare primer containing only zinc chromate with those with both zinc chromate and red lead. The panels were exposed in an acid atmosphere. Zinc chromate is known to stimulate corrosion under highly acid conditions.[11, 30]

Large Scale Field Tests

It is appropriate now to point out that the cost of the program includes many operations besides the application of paint to the panels and the observation and reporting of results. For a paint testing program to be successful it must be complete.[31]

After the durability of the paint system has been established in the panel testing program, it is time to proceed to the large scale field test (equivalent to the pilot plant stage of chemical research). It is at this stage of the development that it is necessary to evaluate the application characteristics, adhesion to old finishes, drying under field application conditions, and finally, actual durability of the system under all conditions prevalent in the plant.

It is possible to determine now the applied cost of a new paint system: The applied cost per square foot divided by the length of time before recoating is the SERVICE COST.[32] The service cost (cost per unit of area per unit of time—cents/square foot/year) should be the final criterion on which selection of a paint system is made. This cost eliminates the need for comparison of such non-comparable costs as the cost per gallon of the paint and the cost per square foot applied.[33]

Figure 5 shows an example of one large scale test. Other large scale tests may vary in size to cover complete tanks and structures. It is at this stage

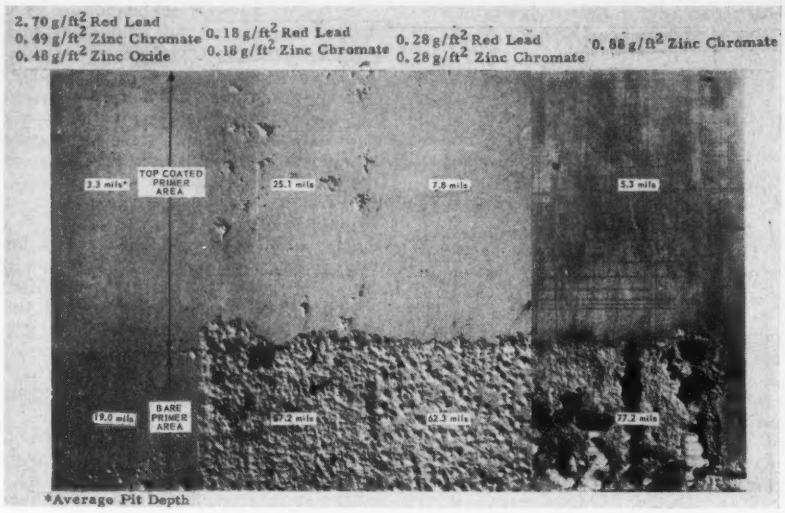

Figure 4—Pitting under zinc chromate-containing primers.

that frequently short-comings of a system are discovered that could not be evaluated in the panel screening stage. Lifting of the old paint being recoated, wrinkling of the new system or one of its components, poor inter-coat adhesion, greater than expected susceptibility to moisture during application or subsequent drying are just a few of the difficulties that may be encountered. It is very important to have capable, well informed applicators for this large scale testing stage. Application is one of the variables which is controlled in the panel testing; it also must be controlled now because the aim is to evaluate a paint product and not the applicator.

Education and communication, probably the most difficult jobs of the corrosion engineer, often overshadow other parts of his responsibility. He must have an open mind and be willing to learn. Most of all he must be willing and able to convey what he has learned throughout his company—from management to painter. Formal reports bulging with economic data and actual photographic evidence to confirm conclusions are adequate in communicating to management but a less formal and more personal approach is necessary with the applicator. It is here that most of programs have their greatest weakness because the applicator either is assumed to be psychic or his function is ignored as being unimportant. Actually his function is one of the most important and he should be fully informed as to what is expected of him and the product he is about to apply. Seagren[34] has stated the problem quite well as follows: "The greatest bottleneck to improve paint performance is lack of knowledge at the application level."

Once a paint system has been thoroughly evaluated through both the panel and large scale tests and found satisfactory, it becomes a product acceptable for purchase and use.

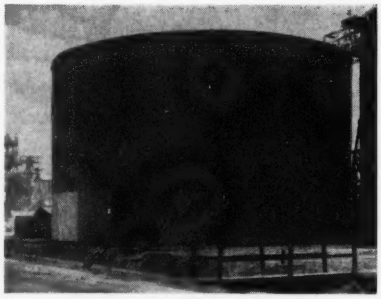

Figure 5—A large scale paint test.

Conclusions

In this article a paint panel testing program has been outlined. A hypothetical chemical plant was used to illustrate some of the economics involved. The statistical design and evaluation of this program was described. Pitting was used as an example of a quantitative dependent variable. Among conclusions the most important ones are:

1. Management must first recognize paint maintenance expense as an item amenable to reduction by a systematic research program.
2. The size of the paint maintenance research program should be a function of the size of the paint maintenance expenditure.
3. A paint screening program using small panels is the most economical.
4. Good experimental design of each group of panels results in data which can be efficiently and economically analyzed.
5. Panel evaluation with several trained observers is essential for proper statistical evaluation.
6. Machine processing of the panel evaluation data gives reliable conclusions in a minimum time.
7. The panel program must be followed by large scale field testing to completely evaluate the paint systems.

8. Education and liaison is probably the most important function of the corrosion engineer.

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Any discussion of this article not published above will appear in the December, 1959 issue.

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Methods for Investigating the

CHARACTERISTICS of REACTIVE COATINGS*

By MAX KRONSTEIN

Abstract

Characteristics of iron, zinc and manganese phosphate treatments are described. Methods are given for preparing standards whose content of reaction coatings can be used for comparison in determining the weight of reaction coatings on specimen coupons, through the use of emission spectroscopy. The role of the so-called metal accelerators is defined. Greater thicknesses can be obtained by using ultrasonic waves, especially on parts of intricate cross-section.

Data are given for zinc and manganese conversion coatings, and tests are described whereby the thicknesses of these coatings can be determined with a high order of accuracy. A method of determining volume of conversion coating through use of the decreasing resistance of a wash water is described.

Investigation showed that the function of a dichromate sealer is to release chromate ions, which are inhibitive. Coatings containing chromic compounds apparently have no inhibitive effect.

The investigation showed that a reaction product formed underneath organic coatings on steel and that it differed considerably from the composition of the paint. The amount of this reaction coating is small compared to that produced by liquid phosphatizing solutions and there was evidence that chromates in the paint are the active inhibiting agents. Another type of reaction not involving liquid phosphoric acid solutions is described for oleo-resinous paints. 5.9.4

Introduction

REACTIVE coatings are surface applications which produce a chemical inter-reaction with the metal surface to which they are applied. Such coatings are either inorganic surface treatments, such as phosphatizing solutions, chemical pickles or sealer treatments, or pigmented organic coatings with reactive components. In all cases an interface layer is formed on the metal surface which is the result of a chemical interreaction between the substrata and the applied treatment. This article is concerned with these reactions and methods for their investigation.

The most important group of inorganic reactive coatings is that of the so-called phosphatizing treatments. While these treatments are applied extensively throughout industry, interpretation of the reaction is based mostly on theoretical considerations. It is interesting that while the patent literature on phosphatizing solutions shows a great number of interpretations for the reactions which are supposed to occur in their application, it does not give any methods for their determination.

These coatings generally are divided into iron phosphate treatments, zinc phosphate treatments and manganese phosphate treatments. Basically the second and third treatments differ from the first by the fact that the iron phosphate solutions initially do not necessarily contain any iron phosphate; the two others contain considerable amounts of zinc or manganese phosphate. In application the iron phosphate is formed during the spraying of the initial alkali phosphate solution on to the steel from which iron phosphate compounds enter the circulating spray solutions from the "run off" in the spray process. In the case of the two other types of treatments, zinc or manganese phosphates are contained in the initial solutions and zinc-iron phosphates and manganese-iron phosphates are formed. During application they enter the circulating system in a similar manner.

Another basic difference between the iron phosphatizing solutions and the two other treatments is that iron phosphatizing solutions contain activating components which are not necessarily present in the two other groups. These are an organic component, such as from the group of sugars or tannins, and a so-called "metal accelerator," such as the anhydride of molybdic acid,[1] tungstic acid, or others. Zirconium compounds have been mentioned also. The part which these components play in the coating formation is not clearly understood. Most studies reported here are concerned with iron phosphates because they offer a better opportunity to examine the chemical changes which take place since they do not contain amounts of metal phosphates large enough to overshadow the new formations.

For these studies test solutions were applied by pressure spray, using usually 14 to 15 psi at 160 to 180 F, giving each side of a 1010 steel panel a one minute spray time. The resulting coatings then were investigated in order to study the reaction products.

Technique of Removing Coatings

In studying the reaction coatings after their application it is necessary first to separate them from the base on which they have been formed. Otherwise the weight of the very thin coating layer would be so small compared to the weight of the substrata that variations in the reaction would be very difficult to identify. So the resulting surface coatings were removed by immersing the panels for 45 seconds in a solution of 300 ml formalin (39%) and 600 ml hydrochloric acid (37%). The amount of the dissolved matter was determined as weight difference of the panels before and after immersion in this "stripping solution." The fluid which contained the dissolved reaction coating, then was concentrated by evaporating most of the water, hydrochloric acid and formaldehyde until in all cases the dissolved matter was contained in less than 40 ml of fluid. By the addition of water an accurate 40 ml level was then obtained.

Rotrode Technique Used

These solutions were subjected to an analysis by emission spectroscopy, using a 1.5 meter spectrograph, and sparking the test solution by the Rotrode technique in an excitation attachment. In order to obtain the same comparative base for all investigations and for the evaluation of the spectrum films, each test specimen was combined with a background material. This consisted of the addition of 10 ml of gold chloride solution (containing 8.2325 mg/ml gold chloride) to each 40 ml of test fluid. As a result each complete test solution consisted of 50 ml of fluid, which contained the stripped matter together with the background material.[2] Density of the resulting lines in the films of the spectrometer was compared, using a film comparator-densitometer.

To begin with three calibration solutions were prepared which contained known amounts of each of the expected components of the reaction coating. Calibration curves obtained from 200 ml of these solutions permitted obtaining from the measurements density values corresponding to the milligram content of each component in 200 ml of solution. (See Figure 1—The Calibration Curves). A control application of the three standards preceded each spectrograph test series of four specimens on the same film strip.

In order to compare the 200 ml reference information with that obtained from the 50 ml solutions, their data were divided by four. This permitted comparison in milligrams of stripped matter.

About the Author

MAX KRONSTEIN—Senior research scientist in the Research Division, College of Engineering, New York University. He was born in Basle, Switzerland, studied at various universities in Holland and Germany and received his PhD in chemistry in 1922 from the University of Leipzig. He then entered industrial work; first in Germany and since 1939 in this country until he joined New York University in 1946. Since that time he has been in charge of research projects in the field of surface chemistry and paint technology which have been sponsored by various branches of the U. S. Armed Services and by private industry. He has published approximately 30 papers in the field of his work.

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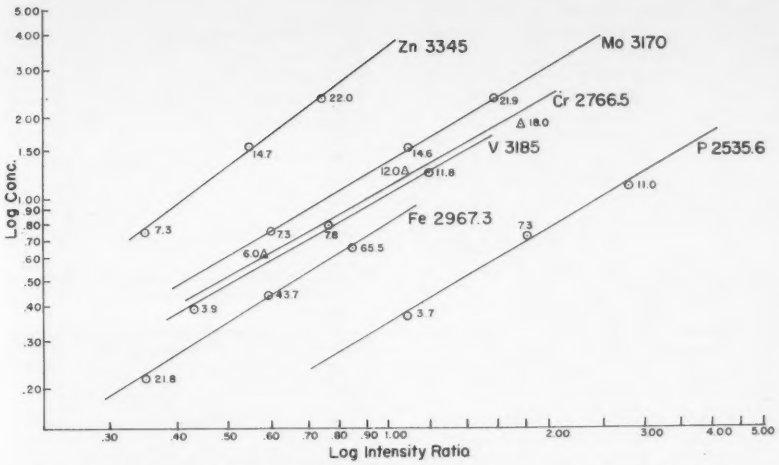


Figure 1—Plot of reactions with standard solutions No. 1A, 2A, 3A. Internal Standard: Au 3029.2.

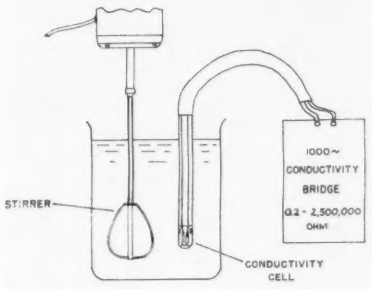


Figure 2—Diagram of setup for conductivity measurements.

From these data, by calculation, the percent content of this matter in 100 mg can be determined.

By measuring the area from which the stripped matter was dissolved, it is further possible to calculate how many milligrams of reaction coating had been formed on a square foot of surface, and how many milligrams of phosphorus and iron or zinc or of the components of the applied metal accelerator had become part of the surface coating in a square foot. An example of such a calculation is given in Table 2.

From the large number of tests made in this manner, a number of facts have emerged.

These may be described as follows: The chemical process of phosphatizing steel is a process of inter-reaction between the applied spray solution and the sprayed steel surface. In this reaction the various metals which are present in the spray solution become a part of the reaction coating. Also, the so-called "metal accelerators" not only accelerates the formation of an iron phosphate; but also becomes a part of the reaction coating. This makes it essential that satisfactory amounts of metal accelerators be used. Where these have a limited solubility in the spray solution, the quantity may be increased by chemical action.[3] The role of the organic matter in the formation of the reaction coating is still being investigated, but it has been established that its structure influences the coating formation.[4] It has been found that the amount of reaction obtainable in coating thickness due to the application of the iron phosphates is not necessarily limited to the low weights now often found. Basic investigation indicates that a considerable increase in the reaction effect can be developed in the future. Finally, the inter-reaction between a steel surface and a phosphatizing solution can be influenced further by removal of the air cushion on the reaction surface and by other physical effects resulting from an exposure of the surfaces in the phosphatizing solutions to ultrasonic waves. The increased coating effect appears as an increase in stripping weight and as successful coating of irregular surfaces, such as the threads of screws, etc. which otherwise would be difficult to react uniformly.

TABLE 1—Example of a Calculation of the Composition of a Stripped Iron Phosphate Coating

Type: Iron phosphate. Organic compound: Dextrose. Metal accelerator: Molybdic anhydride. Stripped from 4 panels: 41.1 mg. Calculated stripping weight: 96.23 mg. per sq. ft. surface.

A. Result of the Spectrum Analysis

	Against 200 ml. Standard Solution, mg.	Per 50 ml. Test Solution, mg.
Molybdenum	31.5	7.875
Iron	36.3	9.075
Phosphorus	5.5	1.375

B. Calculated Composition in Percent Found in 41.1 mg.

	Stripped Matter, mg.	Per 100 mg. Percent
Molybdenum	7.875	19.16
Iron	9.075	22.08
Phosphorus	1.375	3.345

C. Calculated Coating on One Sq. Ft. Surface

	Percent	mg
Total stripped coating on sq. ft.		96.23
Molybdenum	19.16	18.45
Iron	22.08	21.25
Phosphorus	3.345	3.22
Total	44.6	42.92*

* Remainder oxygen, hydrogen, etc.

Application to Zinc Phosphates

The spectroanalytical method of investigation can be applied to zinc phosphates also. As a result of the much higher volume of strippable coating, concentration in the stripping solutions is much higher and would require different calibration solutions for analysis. This can be avoided by further diluting the test solutions by the addition of water and by increasing to the same extent the gold chloride background in the solutions. Results in Table 2 clearly indicate that zinc phosphate also produces reaction coatings with steel and that the resulting coating can be described as zinc-iron phosphates. The method is sensitive enough to identify the elements of chemical additives to the base in the resulting coating. Corresponding tests on manganese phosphates have not been made yet, but it may be expected that the methods are applicable to them also and that they produce chemical reaction coatings also.

The extent to which the water-sprayed solutions become insoluble materials on the steel surface can be studied by electrical conductivity measurements. This is done by immersing the treated surface in distilled water of measured electrical resistance and by measuring the change in resistance, or the increase in conductivity of the solution as a factor of the time of immersion. (Figure 2.) Because a chemical reaction coating is not a coherent organic film, water will penetrate it and reach the steel surface underneath from which water soluble matter will enter the water. The resulting drop in resistance is proportional to the integrity of the coating on the treated surface.

Figure 3 shows resistance drop test of a surface produced by immersing the steel for two minutes in hot phosphoric acid. The so-called iron phosphate surface is so water soluble and the dissolved matter so conductive that resistance dropped from 100 percent to 0.2 percent in 12 seconds. Figure 4 shows results of four resistance tests, one of an iron phosphate and the other of a zinc phosphate surface where very small amounts of soluble matter were immediately available. Results are given also of zinc phosphate and iron phosphate surfaces from which larger amounts of soluble matter caused a marked drop in resistance during the first minute of immersion. More details on these conditions have been reported earlier.[5]

It is possible to remove some of the water soluble matter by a 30-second water rinse after the usual chromic rinse and before the paint application. This influences corrosion resistance of the subsequently painted system under salt-fog exposure. One such example is shown

TABLE 2—Application of the Emission Spectroscope Method to Zinc Phosphate Coating

Stripped matter, total	859.6	mg
1/10 of stripped matter used in the diluted solution	85.96	mg
Stripped area	1.062	sq. ft.
1/10 of stripped area, covered by the matter in the diluted solution	0.1062	sq. ft.

ANALYSIS

	In 200 cc (Volume of Standards)	In 50 cc (Solution)	Per 100 mg Coating	Per Sq. Ft.
Phosphorus	15.8	3.725	4.333	35.075
Chromium	not detected	—	—	—
Iron	19.3	4.825	5.613	45.433
Zinc	150.0	37.50	43.508	353.107
Total				433.615

(Determined stripping weight: 739.7 mg/sq/ft)

in Figure 5 where the 30-second rinse after the usual 30-second chromic rinse (pH 4.5 at 80 C) increased the saltfog life of coating on the painted panel. A longer rinse removed some of the useful deposits of the chromic rinse. These records are taken with the electrographic printing method, described earlier.[6]

Application to Light Metals

Different test methods have been developed for the study of the dichromate sealer application, one of the most important chemical reaction treatments for light metals, especially magnesium. In this application the pickled FS-1 magnesium is immersed for 30 minutes at 200 F in a water solution of sodium dichromate and calcium fluoride and is then rinsed with hot and cold water. This treatment produces a leveling out of the surface profile of the pre-pickled metal surface, either by dissolving the peaks or by depositing the sealer solution or by a combination of both. This is shown in Figure 6.

To study this question the diffraction x-ray spectrum was taken on both the unsealed and sealed surfaces. Figure 7 shows the extensive chromium peak which has been developed by the application of the dichromate sealer.[7] This indicates the treatment results in formation of another reactive coating.

Nature of Coating Investigated

It was further of interest to know whether this reaction coating is based on a chromate compound or on a chromic compound. This is important because chromate compounds can be expected to have a corrosion inhibiting effect under conditions where the chromic compounds do not have such an effect. The chromate ions were identified by immersing the treated panels for extended periods in water and detecting freed chromate ions by the color effect on diphenyl carbazide. The degree of the color reaction was determined by transmission readings. Most of the soluble chromate ions were freed during the first 21 hours of immersion, less during the next 124 hours, and considerably less in a third period of an additional 191 hours. If a chromic coating compound is immersed under the same conditions, no color effects can be detected with the same indicator.

These tests showed that the dichromate sealer application is another chemical reaction treatment and that the resulting coating is capable of releasing chromate ions. The rate of the release under immersion would be considerably less through a paint film. Ordinarily, the supply of chromate matter would remain available for a much longer period and the inhibiting effect of the reaction coating would be much longer.

Tests have not been made yet on painted surfaces.

Other Reaction Coatings Probed

The work on inorganic reaction coatings has produced a number of new methods of investigation which make it possible to investigate reactions of pigmented organic coatings also. Earlier research has been limited to a study of the reactions which occurred inside the fluid and not to those in applied paints.

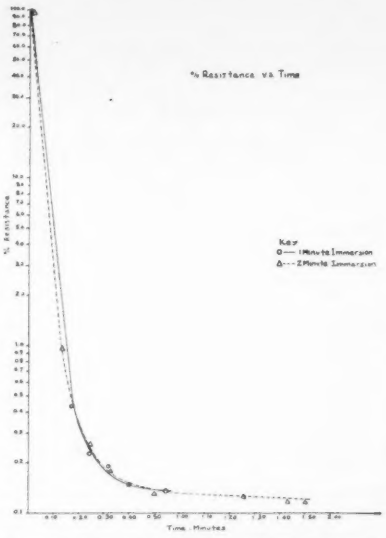


Figure 3—Plot of decreasing resistance of wash solution influenced by products leached from a coating produced by two minutes' immersion of steel in hot 85 percent phosphoric acid.

The most important characteristic of reactive primers is their reaction with the metal surface. Because work on inorganic reactions has shown that a steel surface is much more reactive than often has been assumed, it is to be expected that a phosphoric acid solution inside an organic coating—as is the case with the wash primer—actually would produce not only reactions inside of the fluid paint but also on the metal surface.

Because the inorganic components of the wash primer are the zinc and the chromium of the pigment and the phosphoric acid of the additive, it was to be expected that the inter-reaction product would consist of zinc, chromium, phosphorus and iron, the latter as a result of the inter-reaction. (Table 3). Experience has shown that the reactivity of the wash primer coating decreases within eight hours after the addition of the phosphoric acid solution. To investigate this reaction, the paint was applied to cleaned steel surfaces and was allowed to dry for a reasonable time. Then the organic matter was washed off with ketone mixtures and the surface was cleaned further by mild rubbing off of adherent paint matter with a surplus of solvent. The reaction coating formed underneath the paint as a surface reaction product on the steel was thus exposed. Subsequent analytical data clearly indicate that the paint film was removed, because the composition of the remaining reaction coating differed considerably from that of the paint.

TABLE 3—Components of a Wash-Primer

	Parts
Vinyl butyral resin	56
Zinc oxychromate	56
Magnesium silicate	8
Butyl alcohol	125
Ethyl alcohol	380
Added Before Use:	
Phosphoric Acid	28
Water	25
Ethyl Alcohol	102

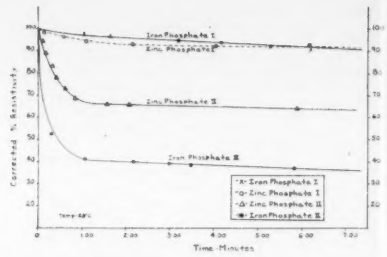
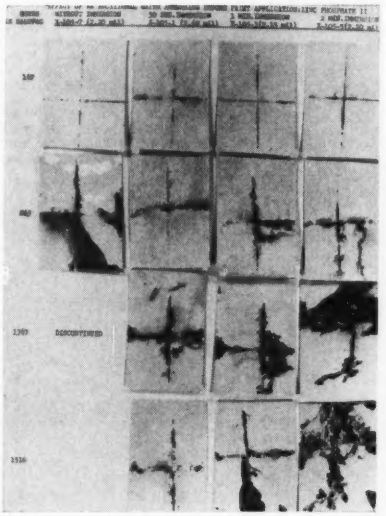


Figure 4—Solubility tests of conversion coatings corrected for percent of resistivity vs time.



1 2 3 4

Figure 5—Effect of additional water rinse before paint application over zinc phosphate conversion coating. Results of salt fog exposure for times indicated versus varying times of water rinse. Column 1—No immersion. 2—30-Second immersion. 3—One minute immersion. 4—Two minute immersion.

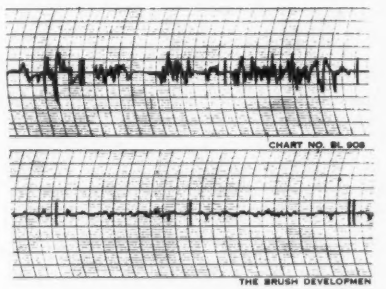


Figure 6—Influence of dichromate sealer application on surface profile of pre-pickled FS-1 magnesium panel. Using Brush Surface Analyzer at setting 0.01. Top: Pickled panel without sealer. Bottom: Pickled panel with sealer. Sealer composition in parts: Sodium dichromate, 570; calcium fluoride, 8.6; wetting agent, 1; water, qs one gallon. Application: immersion 30 minutes at 200 F; followed by rinses, 30-second cold water, 15-second 175 F water, oven dried.

Amount Produced Is Smaller

Results of the analysis (Table 4) indicated that the amount of the "strippable" reaction coating which resulted from application of the wash primer was considerably less than that produced by a spray application of an inorganic phosphate coating. However, the surface coated with the reactive primer was

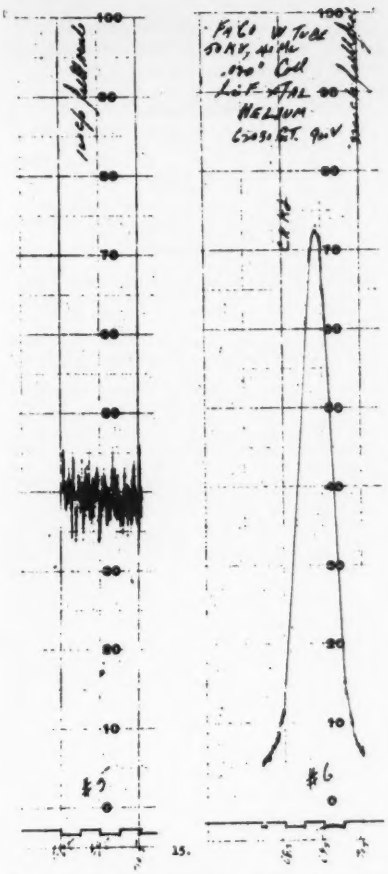


Figure 7—X-ray spectrographs of the pickle and sealer applications on magnesium. Chart 5, surface after pickle; Chart 6, surface after sealer. Interpretation: No. 5—100 c/o full scale (multiply number of chart squares by 1); No. 6—3200 c/o full scale (multiply number of chart squares by 32).

covered by a reaction coating which consisted primarily of a zinc-iron phosphate.

Because the amount of chromium in this reaction coating is low, as shown in Table 4, it was desirable to study further the part which chromate plays in the coating formation. So a different wash primer was prepared in which the zinc oxychromate was replaced by the same amount of zinc oxide. Again a chemical reaction coating was obtained on the steel, but the amount of strippable coating was considerably lower than that formed in the presence of chromate (Table 5), and the ratio between zinc and iron was changed. The data show that the coating contained considerably less zinc than was the case with zinc oxychromate pigmentation. This indicates that the chromate is an activator in the coating reaction.

While making these tests, it was observed that a zinc oxychromate wash primer, after having been in contact with the phosphoric acid solution for three days, no longer produced a reaction coating. This is in agreement with the empirical findings of the industry that the wash primer has a limited "life" after the phosphoric acid solution has been added.

The question remained whether or not the only form of reactive coating produced by paints depends on the addition of phosphoric acid solutions. If this be the case, it would be very difficult to produce reactive paints based on oleoresinous materials.

Other Reactions Are Possible

But it is evident that other formulations can be developed such as introducing a phosphorylation group into a soybean lecithin. This can be done by treating the lecithin with an organic per-acid, with a subsequent phosphorylation with phosphorus pentoxide. The resulting product retains its phosphatide molecular structure without charring under the treatment. The surplus of free acid can be neutralized with an alkali.[8] First indication that this material acts as a reactive medium was observed when SAE 1010 panels were immersed in a solution of one part of this reaction product in six parts toluol (Table 6). The resulting surface modification produced a 36 percent decrease in surface reflectance of the steel surface as compared to those of similar panels immersed in plain toluol.

That a strippable coating is produced has been established further by determining the stripping weights of the coatings. These stripping weights are considerably higher than those obtained from wash primer applications and clearly indicate that the phosphorylation group of the organic lecithin can react with the steel without addition of a fluid phosphoric acid solution.

The question remained whether or not so-called inhibiting primers without any phosphoric acid or phosphoric acid

TABLE 6—Reaction Coatings Produced From Phosphorylated Lecithin Solutions*

Solutions prepared from phosphorylated lecithin and toluol 1:1.
SAE 1010 steel panels immersed for 2 minutes, followed by exposure in a drying oven at 300 F.
Tests made with different preparations using different neutralizers for excess acid.

	Hours In Oven	Wt Stripped Coating, mg	Stripping Wt mg/sq. ft.
Solution A	1	7.7	64.0
	3	7.9	63.7
Solution B	1	8.0	66.0
	3	9.2	75.8
Solution C	1	10.3	84.1
	3	9.1	83.5
Solution D	1	10.5	84.0
	3	12.8	107.9

Coating effect of a phosphorylated lecithin-toluol solution (ratio 1:6) can be measured as decrease in surface reflectivity (decrease 36%).
Effect as paint additive has been developed further.

* Lecithin treated with an organic peracid, followed by phosphorylation with phosphor pentoxide. U. S. Patent 2,839,545.

TABLE 7—Inter-Reactivity of Zinc Chromate Primer With Iron Phosphate Reaction Coating
(Preliminary Data)

PAINT COMPOSITION

	Grams
Zinc chromate	350
Alkyd resin	380
Mineral spirits	91
Cobalt naphthenate 6%	1.2
Lead naphthenate 24%	0.75
Calcium naphthenate 5%	0.75
Viscosity 1010 cps Diluted before application with mineral spirits	37

APPLICATION

a. Applied to an iron phosphate coating the reaction film of which had been analyzed and which contained no zinc.
b. Film thickness of the applied paint film: 5.3 mils.

STUDY OF THE STEEL SURFACE AFTER PAINT REMOVAL

	mg/sq. ft.
a. Stripping weight of the unpainted phosphatized panels	96.233
b. Stripping weight of the panels after painting and paint removal	151.4

ANALYSIS

Zinc content of reaction coating before painting: none.
Zinc content after painting and paint removal: 3.55%.

TABLE 4—Identification of the Reaction Coating Produced on 1010 Steel by Application of the Zinc Tetraoxychromate-Polyvinyl Butyral Primer

METHOD

a. Repeated application and washing off of the paint (time between application and washing off: 10 minutes)
b. Stripping of the chemical reaction coating from the panel, for example.

	mg/sq/ft
Stripping weight after 3 applications	46.1
after 4 applications	63.5
after 5 applications	64.0
after 6 applications	67.0
Average	60.15

Total dissolved from the four panels: 42.5 mg

ANALYSIS OF THE DISSOLVED MATTER

	200 ml Solution, mg	50 ml with Stripped Matter, mg	CALCULATED % 100 mg	CALCULATED mg sq. ft.
Iron	22.5	5.625	13.22	7.95
Zinc	31.0	7.75	18.23	11.765
Phosphorus	6.7	1.675	3.95	2.376
Chromium	1.2	0.3	0.7	0.421

Comment: Similar results were obtained with shorter contact time between paint and steel surface, such as 4, 6 or 8 minutes.

TABLE 5—Replacing the Pigment in a Wash Primer With Zinc Oxide

PAINT COMPOSITION

Parts	Added Before Use.	Parts
Vinyl resin 56	Phosphoric acid	28
Zinc oxide XX 505 54	Water	25
Magnesium silicate 3X 8	Ethyl alcohol	102
Butyl alcohol 125		
Ethyl alcohol 380		

APPLICATION

Four coats at 10 to 15 minute intervals on 6 panels. Average stripping weight. 28.5 mg/sq. ft.
Average stripped matter: 30.6 mg

ANALYSIS

	200 ml Solution	50 ml with Stripped Matter	CALCULATED % 100 mg	CALCULATED mg sq. ft.
Phosphorus	3.8	0.95	3.104	0.884
Chromium	not detected	—	—	—
Iron	15.5	3.90	12.745	3.632
Zinc	8.2	2.05	6.700	1.909

CALCULATION OF THE AMOUNT OF METAL TO ONE PHOSPHORUS

	Zinc Chromate Wash-Primer (Average of 4 Determinations)	Zinc Oxide Wash-Primer
Iron	3.184	2.919
Zinc	3.344	1.318

groups also might react chemically with the surface. There are not enough data now to decide this question, but one interesting test series is indicative. SAE 1010 steel panels having an iron phosphate coating of determined composition were painted with a zinc chromate alkyd primer (Table 7). The organic coating was then removed in the manner described earlier and the chemical reaction layer of the earlier phosphatizing treatment exposed. In order to determine if this layer had changed its composition as a result of the coating with the zinc chromate primer, the chemical coating was stripped off. Its weight per square foot had increased from 96.233 mg/sq/ft to 151.4 mg/sq/ft. Analysis of the earlier phosphate coating did not show any zinc but the stripped phosphate coating afterwards showed 3.55 percent zinc. This indicates that the zinc chromate primer did inter-react with the phosphatized surface.

Summary

In summary, methods have been developed and applied to the investigation of reaction coatings as formed by application to metal of inorganic chemical surface coatings and by pigmented reactive organic coatings.

It was found that these types of coatings produce a chemical modification of the substrata and that it is possible to analyze these coatings by spectroanalytical methods.

It is possible also to control and to modify systematically their formation.

These reactions are of great importance for corrosion protection, because earlier work[9,10] clearly establishes that surface reaction coatings when used as a base for organic coating systems increase considerably the life of the complete system under saltfog exposure and under other evaluation tests.

Acknowledgments

The author expresses his appreciation to J. E. Van Dien of the Applied Spectrochemical Laboratories, Glen Rock, N. J. for his cooperation in the development of the tests reported in this article.

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Any discussion of this article not published above will appear in the December, 1959 issue.

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Analysis of

Industrial Roof Construction and Maintenance*

By J. C. LEMMON and W. G. CRAIG

Abstract

Discusses the factors that govern the costs of built-up roofing during the life of the building. Limitations of construction and surfacing materials are given. Maintenance methods and materials are explained and a program for securing maximum utilization of roofing expenditures is presented.

8.8.1

Introduction

SERVICE LIFE of industrial roofing depends upon the quality of construction materials, workmanship and methods of construction and environment to which the roof is exposed. The adverse environmental factors are, in the order of their importance, ultraviolet light, oxygen, moisture, temperature, wind and contaminants in the industrial atmosphere. Effect of the first four factors on various materials of roof surface construction, patterns of the resulting deterioration and materials available for preventive maintenance and repair have been published.[1]

Ultimate cost of industrial roofing depends upon how well the original specification selection and maintenance procedures are coordinated to obtain an effective balance between capitalized investment and tax deductible maintenance dollars.

The whole problem requires a basic engineering knowledge of materials and methods and an understanding of fiscal matters as they relate to the maximum utilization of capital.

Principles of sound roofing can be applied to a variety of materials and types of construction. In this article, these principles will be applied to the most widely used form of industrial roofing—the so-called built-up roof. This is a highly descriptive term: these laminated structures are built up on the roof deck by the application of multiple layers of reinforcing membranes (felts) and adhesive waterproofing bitumens installed over roof decks which can be of many types of construction and of any slope from dead level to more than 45 degrees. Most industrial structures feature the dead level or very low pitch construction which complicates the waterproofing problem. Water is often held on the structure rather than shed from it, and prolonged exposure to moisture is one of the major causes of deterioration.

Roofing has only one function—preventing the penetration of water. In a built-up roof this waterproofing is a function of the bitumen, not of the felts, because all felts are intrinsically water-absorbing and are deteriorated by this moisture absorption. Base felts, which must be somewhat pliable and capable of some elongation to adjust to deck movement, are used principally to strengthen the final structure and to divorce the surface waterproofing from building stresses which might fracture it. Consequently, all maintenance activity must be directed toward preserving the water-proofing top surface; once this surface is breached and water gets into even the topmost felt, the problem is no longer one of maintenance but of replacement of mechanical strength.

Structural Problem

The first engineering problem presented in the construction of a built-up roof over typical wood, metal or concrete roof decking is a structural one (Figure 1). Normal movement, deflection or distortion of decking members will rupture the waterproofing unless ample provision is made to distribute these stresses. Stress distribution is accomplished by installing a heavy roofing felt or insulation board over the decking and by nailing or fastening it to prevent windlift. The felt should be loose enough to allow the deck to move under it as freely as is possible so that this movement cannot rupture the waterproof surface to be placed over it.

The usual practice in built-up roofing is to laminate two plies of lighter felts to this base and to each other with a waterproof bitumen adhesive. This increases the strength of the structure and develops a smooth, stress-free surface to receive the final waterproofing.

Up to this point, construction of all built-up roofing is the same. Properly applied, this construction is adequate to distribute average roof deck stresses under normal conditions of use. Protection against disintegration of this structural base is provided by the surface waterproofing applied over the base. Differences in service life (see Figure 1) and cost of the complete roof will depend upon the nature and quality of the surface waterproofing employed.

A minimum specification (Ten Year Roof) is a simple flood coat of asphalt over the structural elements.

A heavier flood coat of bitumen, top-dressed with slag or gravel for protection from the deteriorating effect of exposure to ultraviolet rays, will up-grade the specification to a fifteen year expectancy.

An additional ply of felt surfaced with a heavy coating of bitumen and slag or gravel will extend basic construction service life to twenty years. Although the additional ply of felt is not required for structural reasons, it does add strength and stability and delays penetration of destructive moisture to the underlying supporting felts.

Service life of the above specifications will depend solely on how long the structural elements of the built-up roof are protected from moisture penetration, which will deteriorate them to the point where they no longer have adequate strength to perform their function. When this occurs, there is no possibility of repair, the only alternative is to remove the old roofing and start over. Cost of this removal must be included in computing the cost per square per year of service for any specification. If insulation is included in the specification, its cost must also be included because there is no possibility of removing the built-up roof without destroying the insulation.

These 10- to 20-year time denominators are based upon estimates of the time before the roof deteriorates completely and removal and re-roofing are required. The guarantees or bonds commonly asso-

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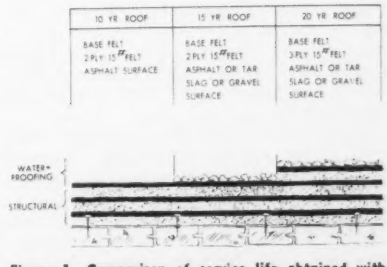

Figure 1—Comparison of service life obtained with different waterproofing in built-up roof construction.

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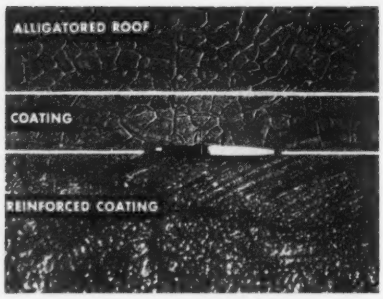

Figure 2—Random reinforcing prevents transfer of crack stress.

TABLE 1—Built-Up Roof Cost Comparison: Replacement vs Maintenance

TYPE OF ROOF	COST OF ROOF					COST OF MAINTENANCE	YRS SERVICE	COST per SQUARE per YEAR
	INSULATION	STRUCTURAL	WATERPROOFING	REMOVAL	TOTAL			
10 YR	0	$10.-	$ 2.-	$5/2*	$14.50	0	10	$1.45
	$10.-	7.50	2.-	8/2*	23.-	0	10	2.35
15 YR	0	10.-	6.-	6/2*	19.-	0	15	1.26
	10.-	7.50	6.-	8/2*	27.50	0	15	1.83
20 YR	0	10.-	8.-	8/2*	22.-	0	20	1.10
	10.-	7.50	10.-	10/2*	32.50	0	20	1.50
10 YR	0	10.-	2.-	0	12.-	$16/2*	20	1.-
	0	10.-	2.-	0	12.-	24/2*	30	.80
	10.-	10.-	2.-	0	12.-	24/2*	30	1.13

*TAX DEDUCTIBLE ITEM. REAL COST APPROX. 50% OF ITEM

ciated with built-up roofing specifications offer limited financial protection and often interfere with the proper timing of maintenance because the tendency is to wait for the bond period to expire before taking remedial action. It is then usually too late for anything other than tear-off and replacement.

A comparison of the cost of maintenance versus replacement is given in Table 1, which shows the approximate applied costs of various specifications. (Where insulation is used, cost of the *structural* portion of the roofing specification is less because the insulation serves as a stress distributing element, thus a heavy base sheet is not required.)

Cost comparisons in Table 1 were obtained by assuming a given set of conditions and applying them to various specifications to obtain comparable costs per square per year of service. Values shown should not be assumed to apply to any particular location or job; local labor rates, job conditions, size, etc., will vary and affect the actual cost.

Data in Table 1 show that the cost of the structural portion of the built-up roof is the same for each type of construction and that the cost of the final specification varies with the cost of the waterproofing employed over the structural base.

In all cases it has been assumed that structural and waterproofing elements will be exhausted in obtaining the maximum specification service life without maintenance and will have to be removed. Removal costs, however, may be expensed as of the period in which they occur and, therefore, have been divided by two (to reflect the approximate effect of the 52 percent corporation tax) and the result added to the original cost to determine total actual cost. The cost per square per year is this actual cost divided by the specification's years of service.

This comparison reveals that one way to obtain lower roofing costs per square per year is to increase the investment in surface protection of the original specification. A 20-year roof will cost approximately $1.10 per year versus $1.45 for the 10-year specification but involves a 50 percent increase of the investment in unproductive capital assets. Most industries prefer to keep this money in working capital for an expected profit return rather than to tie it up in nonproductive capital assets.

The bottom three lines of Table 1 show that the lowest cost per square per year for built-up roofing can be obtained by making the minimum initial capital investment (a 10-year specification) and then maintaining this roof in good condition by adequate surface care by spending tax-deductible maintenance dollars. More money can be spent in this way than was spent on the roofing originally and actually result in a lower cost per square per year of service than can be obtained by any other means.

A roofing program designed to obtain these lower over-all costs and reduced capital investment features must recognize these fundamentals:

1. Surface waterproofing necessary to protect the investment in the supporting felts must be kept intact.

2. Protection must be provided *before,* not *after,* deterioration of structural elements occurs.

3. Performance characteristics of maintenance materials used must be such that maintenance can be continued over a long span of years.

Gravel and slag surfacings are effective in prolonging service life of exposed bitumens, but they represent increased capital investment and make inspection, repair and visual determination of the condition of the supporting felts difficult. They cost approximately 50 percent more to remove, before maintenance can begin, than they cost to install originally.

Wherever conditions permit, the smooth surfaced asphalt specification represents the lowest initial capital investment and the best opportunities for continuing economic maintenance of the surface waterproofing. A complete understanding of the pattern of degradation of this specification is required to take advantage of its maintenance possibilities.

When a built-up specification has been applied with the correct strength and stress distribution characteristics for the structure which it covers, the deteriorative environmental factors begin their destructive attack. Obviously, the first element to be attacked is the exposed waterproofing layer of bitumen on which survival of the entire structure depends.

Oxidation, catalyzed by ultraviolet light and accelerated by the sun's heat, results in degradation, hardening and shrinkage within the bitumen. This shrinkage creates stresses which must be relieved. As a result, the bitumen cracks in the familiar alligator pattern illustrated at the top of Figure 2.

At the same time that this is occurring, small pin blisters often appear in the waterproofing surface due to the escape of water vapor which was initially in the topmost felt or to the release of water formed by oxidation of the bitumen. Alligatoring and pin blistering can develop to depths which will allow water to enter the felts within three to five years. Up to this time no great loss of strength or leaking has occurred, but once water gets into the felts, microorganisms will attack and deteriorate the fibers which are the strength-giving element. This deterioration is permanent. Contrary to commonly held theories, strength cannot be restored by resaturating the felt with penetrating bitumens.

Obviously, the optimum time to start maintenance of the waterproofing surface is just before the surface has deteriorated to the point where cracks and pin blisters reach a destructive depth. Careful annual inspections enable the maintenance engineer to determine the danger point on any given structure and to take steps to restore the waterproofing surface. Susceptibility to cracking and pin blisters varies greatly between asphalts, and in shaded areas the defects will appear more slowly than in exposed areas.

The mechanics of restoration of the vital waterproofing surface require an understanding of the physical problem involved and the repair materials available. An alligatored roof surface is like any other cracked surface: its stress pattern is transferred to anything placed over it. This is the reason a simple coating of more bitumen gives very limited protection; it is not strong enough to resist these stresses very long.

Originally, the bitumen is sufficiently ductile to accomodate some stress, but as it ages and hardens this quality diminishes. Also, in cold weather it is brittle and less resistant to sharp stresses. The result, illustrated in the center section of Figure 2, is that the alligator pattern is transferred through the coating in less than two years and deterioration of the felt proceeds after a slight interruption. Successive coatings give even shorter service because the depth of the film magnifies the cracking.

First consideration in choosing a material to maintain a typical alligator roof surface, therefore, is that it be sufficiently reinforced to distribute these crack stresses so they will not recur. This is illustrated at the bottom of Figure 2. (After 10 years of service, this section remains substantially unchanged). Addition of fibers or fillers to the coating bitumens is helpful to a small degree, but fibers long enough to do an effective job of stress distribution are seldom encountered in such formulations and even if used would make a compound which would be difficult to apply. The only effective means of achieving the necessary reinforcement is to use a combination of bitumen and a separate reinforcing element.

Reinforcing elements which can be

used skeleton-wise within the waterproofing surface to give it strength are, of necessity, porous structures which can be filled and coated by the bitumen (Figure 3). They are open weave cotton, jute, glass and synthetic organic fiber fabrics or loosely felted mats of the same fibers.

Woven reinforcements, illustrated in Figure 4, leave something to be desired as their strength is high in only two directions. As a result, they have little stretch or yield and therefore, under tension, tend to pull up thru the bitumen and cause pin-holes. Randon fiber mats, on the other hand, have ample strength in all directions coupled with good stretch and yield. Fiber mats conform readily to any irregularities in the structure and stay tightly in place in hollows or swales in the surface without developing tensions that will lift or float them out of the bitumen. Only those fibers which will not rot should be used.

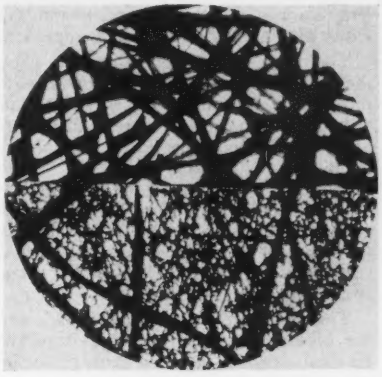

Figure 3—Porous reinforcing materials used within the waterproofing surface.

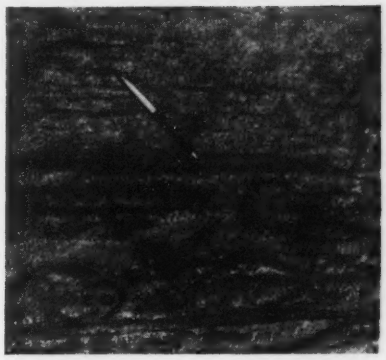

Figure 4—Woven reinforcements have strength in only two directions.

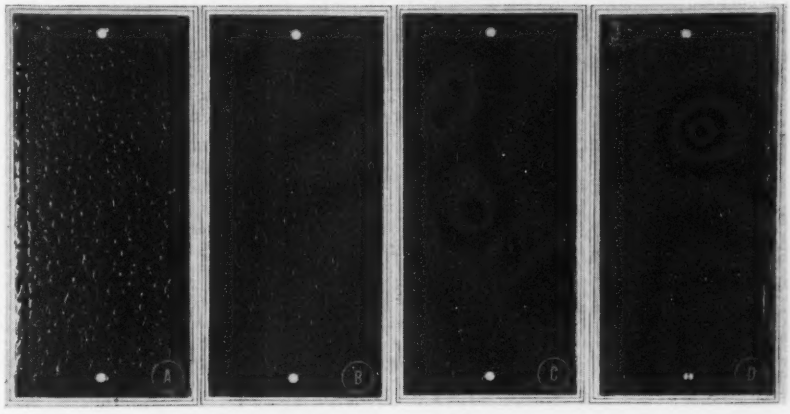

Figure 5—Thin film asphalt weathering tests. Panel A: molten asphalt, 3 cycles; Panel B: cut-back, 3 cycles; Panel C: chemical emulsion, 3 cycles; Panel D: clay-type emulsion, 50 cycles.

Basic Formulations of Asphalt

The second consideration is the nature of the bitumen itself. Almost all smooth surface, built-up roofs are surfaced with asphalt rather than tar because the brittleness and sharp melting point of the latter requires slag or gravel coverage to protect it. Because gravel surfaces do not lend themselves to simple maintenance procedures, only asphalt will be considered here.

There are only four basic formulations for application of asphalt: molten, cut-back (solvent thinned), chemical emulsion and mineral colloid emulsion. With the first three formulations, surfacing left on the roof after the asphalt cools or after the solvent evaporates is a film of asphalt and subject to all of asphalt's variability due to source of origin and method of refining. These formulations can be expected to deteriorate in the same pattern as the original flood-coated waterproofing.

Residue left by the drying of a bento- illustrated in weatherometer test panels in Figure 5, is quite different in its weathering pattern from the asphalt from which it is made. Bentonite gel structure, which forms throughout the asphalt as the emulsion dries, supports and reinforces the asphalt. It also confines the oxidative deterioration of the asphalt to the weather face with the result that the film chalks slowly rather than becoming hard and brittle and shrinking throughout its mass. The result is an exposure durability that exceeds (in this case by 17 times) that of any other formulation of asphalt.

In addition to its great weather resistance, bentonite clay type emulsion residues have the ability to pass water vapor through the film but to block the passage of liquid surface water. This breathing characteristic is important in restoring the waterproof surface of a roof. Some moisture will always be in the cracks and crevices of the old roof. If this moisture cannot escape easily, it will develop sufficient pressure to cause delamination of the new waterproof surface from the old and result in blisters that severely limit performance.

Because of these unique performance characteristics, bentonite clay emulsions in combination with a strong non-rotting random fiber mat reinforcing (to which the emulsion residue is closely adhered) provide an ideal surface waterproofing medium. The deterioration pattern of asphalt clay emulsion is by surface erosion and chalking rather than bulk hardening and shrinkage; therefore, continuing effective maintenance requires only periodic renewing of the surface with a light application of more emulsion without further need for internal reinforcement. Recoating at about seven to ten year intervals appears to be adequate.

Product combinations that will meet all of the conditions outlined for preventive maintenance are available and can be applied to typical roof surfaces for $10 to $14 per 100 square feet. This is well within economic limits shown in Table 1. The effects of economic inflation on removal and re-roofing costs undoubtedly will make these limits even broader in the future.

The same type of maintenance procedure also can be used satisfactorily on roofs that have suffered some strength loss in the top ply. This can be done by using a stronger reinforcing mat and a larger quantity of bitumen for embedding and coating. Cost range for this type of *corrective* maintenance usually will be $16 to $20 per 100 square feet.

Special Problems

So far, only the normal deterioration of roofing has been considered. In certain areas within any industrial plant, the destructive influence of specific chemicals falling or condensing on the roof will greatly complicate the problem. In most cases the problem is so specific that extensive research is required to find the answer. Consequently, there are no pat answers or commercial products readily available to meet all conditions.

Asphalt is highly resistant to inorganic acids except those of high oxidation potential such as nitric and concentrated sulfuric, but it is badly deteriorated by condensation of organic acids or solvents on the surface. In such situations, vent ends of breather pipes and fume ducts should be extended to the side of the roof. If this is not possible, polychloroprene, Buna N, chlorosulfonated polyethylene and other specialty coatings may help, but information on their durability is limited.

Industrial dust accumulation occasionally creates a serious problem. Unless specially treated, bentonite clay emulsions can re-emulsify badly by fall-out of materials with high sodium ion concentration such as sodium carbonate or sodium phosphate. These clay emulsions are not damaged by materials containing divalent cations.

Accumulation of deposits of materials such as dextrins, phenolic resins and other polymerizable materials on the roof surface invariably will result in cracks and splits in the felts. This is due to the shrinkage of these deposits during weathering, which pulls the roof surface with it and tears the felts. Surface coating of such a roof with a highly filled and quick chalking compound will help by presenting a dusty surface to which the deposits will not adhere as tenaciously as to asphalt.

Fortunately, these special problems usually are highly localized and are not as important in over-all maintenance of industrial buildings as is the care of the standard built-up roof under average service conditions.

The biggest engineering mistake in roofing is to allow the expensive structural elements of a built-up roof to be consumed in order to obtain maximum service life of a specification simply because of some arbitrary time denominator that was attached to the original selection, instead of dealing with it as a maintenance problem requiring sound engineering and fiscal analysis.

Roofing and roof maintenance often is poorly administered because various management branches, particularly finance, engineering and purchasing who are involved in different aspects of the problem, do not thoroughly understand the value of a closely coordinated approach.

The biggest fiscal errors occur when the original selection of the roofing specification is not made on the basis of obtaining the necessary structural factors at the lowest possible initial capital expenditure. At the same time, a preventive maintenance program should be organized to obtain surface protection continuity for the structural elements at the first sign of breakdown of the original surfacing and long before leaks occur from structural failure.

This program must recognize the number of different departments and people that can become involved during the life of a typical roof.

The engineer's biggest task is to translate his technical knowledge of roofing into specifications (or standards) which will be practical to use in purchasing and in estimating amortization schedules. This involves specifications that clearly delineate the materials and procedures that engineers consider essential to the solution of a given problem.

The biggest problem for the purchasing department is choice of purchasing standards to be used in asking for bids so that the engineers get the products desired. Simple physical description of components will not provide positive protection against such limiting defects in roof surfacing materials as loss of strength, alligatoring, blistering, flowing, rotting, floating and failure of the components to remain bonded together for prolonged exposure periods.

Protection against these limiting defects can be accomplished best through purchasing specifications that make responsible performance, free of these specific defects, a definite condition of use. Failure of a surfacing specification in any of these important performance areas will severely limit the possibilities of continuous economic maintenance.

Cost control of roofing is chiefly a matter of developing policies, standards and specifications that establish a workable continuity between original selection of roofing specification and maintenance procedures. Necessarily, this must be based on balancing sound economic, fiscal and engineering objectives and on practical recognition of the different persons and agencies involved during the life of a typical roof.

References

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Any discussion of this article not published above will appear in the December, 1959 issue.

HOW CORROSION IS INDEXED

1. Alphabetical subject and author indexes are published in December at the end of the calendar year volume of 12 issues.
2. Abstracts of articles appear in the Corrosion Abstracts section of CORROSION.
3. Abstracts of articles appear in the NACE Bibliographic Surveys of Corrosion.
4. Abstracts of articles appear in the Corrosion Abstract Punch Card Service.
5. CORROSION is Indexed regularly by Engineering Index.
6. CORROSION will be indexed, beginning in January 1958 by Applied Science and Technology Index.
7. A 10-year alphabetical subject and author index (1945-54) has been published and is available for sale.

Corrosion Inhibitor Evaluation from Cathodic Polarization Measurements*

By R. A. LEGAULT and NORMAN HACKERMAN

Abstracts

Investigation described indicates that relative corrosion rates of iron and steel in systems buffered to a pH of 5.0 can be determined from cathodic polarization measurements in the presence of an oil phase, carbon dioxide and commercially used amine inhibitors. Method described provides a rapid and relatively accurate means of evaluating the efficiency of industrial corrosion inhibitors in systems which simulate operating conditions. 5.8.3

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Introduction

THROUGHOUT THE oil-producing industry the methods used most widely for evaluation of corrosion inhibitor efficiency are based on coupon weight-loss determinations. These determinations are made under experimental conditions which simulate, as far as is possible conveniently, actual operating conditions under which the use of the prospective corrosion inhibitor is proposed. These methods cannot be applied to some test media of interest to oil producers because a significant weight loss cannot be obtained in these media without resorting to determinations of relatively long duration. Purpose of this investigation was to provide a rapid and relatively accurate means of evaluating the efficiency of corrosion inhibitors in simulated operating systems.

The theory of mixed potentials[1,2] which has been applied to the corroding Fe electrode[3-7] forms the basis for this study. Primary concern was with problems which confront the oil producer; therefore the relations were studied which exist between the electrochemical behavior of mild steel and its corrosion rate in oxygen-free, acid environments. Among the workers who have studied these relationships, Stern[6] worked with both pure iron and with steel, and Kaesche and Hackerman[7] worked with pure iron while studying the effect of a number of simple amines as corrosion inhibitors.

In these cases, studies were made in media of very low pH. At pH values higher than approximately 2, polarization data appear, despite vigorous stirring of the test medium, to include a contribution from concentration polarization, which is large enough to conceal the meaningful portion of the data. This suggested the use of appropriate buffers for study of media at higher pH values.

The relationship between the corrosion rate of iron and cathodic polarization curves constructed from data obtained on corroding iron electrodes is developed in the next section. Stern showed that this treatment is valid for steel as well as for iron.[6]

When iron or steel is placed in an anaerobic, acidic medium, the reaction involves both dissolution of iron and reduction of hydrogen ions. Thus

$$Fe \rightarrow Fe^{++} + 2e^-$$
$$2H_3O^+ + 2e^- \rightarrow 2H_2O + H_2 \quad (1)$$

Both the anodic and the cathodic processes occur on the one electrode, and the electrode surface may be considered to consist of many local oxidation-reduction cells which essentially control the corrosion mechanism. When the sum of the cathodic process rates equals the sum of the anodic process rates, the potential of the corroding electrode, when measured against a reversible reference electrode, is referred to as the steady state mixed potential. In the system under consideration, it indicates that the oxidation rate of iron equals the reduction rate of hydrogen ions. In terms of current densities, i,

$$i_{Fe} = i_H \quad (2)$$

If an external cathodic current is applied to the polarizing electrode, the steady state potential is disturbed, but a new steady state is usually reached where $i_{Fe} \neq i_H$. Current density of the impressed current is equal to the difference in rate between the oxidation and reduction reactions.

$$i_x = i_H - i_{Fe} \quad (3)$$

When the direction of impressed current flow is such that polarization of the cathodic reaction results, the local action current may be defined as the rate of oxidation of the iron,

$$i_{la} = i_{Fe} \quad (4)$$

and from Equation (3), the rate of reduction of hydrogen ions equals the sum of the externally applied current density and the local action current density.

$$i_H = i_x + i_{la} \quad (5)$$

The Tafel equation expresses the relation between hydrogen overvoltage and current density,

$$\eta = -\beta \log \frac{i_H}{i_{o(H)}} \quad (6)$$

where β and $i_{o(H)}$ are constants. $i_{o(H)}$ is the current density value at the equilibrium potential for the hydrogen reduction reaction and, regardless of its actual physical significance, is called the exchange current; β is the Tafel slope.

Substituting for i_H from Equation (5),

$$\eta = -\beta \log \frac{i_x + i_{la}}{i_{o(H)}} \quad (7)$$

The hydrogen overpotential is the difference between the potential measured with a particular applied current density and the reversible potential of hydrogen.

$$\eta = E_R - E \quad (8)$$

If the potential is measured against a saturated calomel reference electrode (SCE), Equation (8) may be written:

$$\eta = 0.2377 - E \quad (9)$$

Equation (7) now can be rewritten:

$$0.2377 - E = -\beta \log \frac{i_x + i_{la}}{i_{o(H)}} \quad (10)$$

As the value of the externally applied cathodic current density, i_x, is increased, little change in the value of the measured potential is noted until the value of i_x becomes significant with respect to the local action current density. As the externally applied cathodic current den-

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sity is increased, and the point is attained where local action current density becomes negligible with respect to the externally applied current density, the value of i_x becomes equal to the reduction rate of hydrogen ions, i_H. From this point on, the entire electrode functions as a cathode, and plotting the logarithm of the externally applied current density versus the measured potential yields the partial overvoltage curve for the reduction of hydrogen ions. The straight line relationship described in the Tafel equation is now observed, and an equation describing this relationship may be written:

$$0.2377 - E = -\beta \log \frac{i_x}{i_{o(H)}} \quad (11)$$

When there is no external current applied, E becomes the open circuit potential, OCE, which is the corrosion potential, and i_x, when extrapolated to the OCE is i_{is}, the corrosion current density or corrosion rate. Equation (11) can now be rewritten

$$0.2377 - OCE = -\beta \log \frac{i_{corr}}{i_{o(H)}}, \text{ or,} \quad (12)$$

$$\log i_{corr} = \log i_{o(H)} + \frac{OCE - 0.2377}{\beta} \quad (13)$$

where all of the quantities on the right hand side of the equation can be determined from cathodic polarization data.

The part of the curve in the vicinity of the corrosion potential can be obtained by extrapolating the straight line obtained in the cathodic polarization measurements back to the open-circuit potential. The current density at which this straight line intersects the open-circuit potential is the corrosion rate if no change occurs in the corrosion reaction in the section of the curve obtained by extrapolation. Corrosion rates obtained from cathodic polarization measurements made on iron in media of high acidity check well with corrosion rates determined from iron analyses.[6,7] For a more exhaustive treatment of the application of polarization measurements to corrosion processes, a series of papers by Stern should be consulted.[8,9]

The work of Stern[6] and experiments performed during this investigation indicated that attempts to measure activation overpotential of hydrogen in media above pH values of approximately 2 are likely to include an appreciable contribution because of concentration polarization. This phenomenon has been treated extensively in the literature.[6,10-14] King discussed the magnitude of concentration polarization to be expected in the measurement of hydrogen overvoltage.[14]

Calculations similar to those by King indicate that impressing current densities necessary for the construction of the desired polarization curves would result in a very large pH change at the electrode in an unbuffered solution of HCl of pH greater than 2. Impressing the same current densities on the same electrode would result in a negligible pH change in a solution consisting of 0.1N acetic acid and 0.18N sodium acetate in proportions calculated to yield a pH of 5. Use of an acetic acid-sodium acetate buffer to displace the effect of concentration polarization in the study of activation polarization is made even more attractive by the results of a study which showed that the use of acetic acid and sodium acetate with iron does not introduce any adsorption to further complicate the system.[15]

Use of an appropriate buffer will displace the effect of concentration polarization, not eliminate it. At sufficiently high values of impressed current, this effect still will obscure the Tafel relationship sought. Purpose of the buffer is to displace the onset of the concentration polarization effect sufficiently to provide a polarization curve section which displays the Tafel relationship. With experience at evaluating these curves, the onset of concentration polarization can be recognized easily and the straight-line portion of the curve immediately preceding this identified as the Tafel region.

Slope β of this line is calculated by the method of least squares and in turn used to calculate the value of i_o, the current density value at the equilibrium potential for the hydrogen reduction reaction on the SCE scale. These two quantities then are used in Equation (13) to obtain a corrosion rate expressed in terms of current density. Examination of this equation shows that an increase in i_o or in the OCE will result in an increased corrosion rate; an increase in Slope β will result in a decreased corrosion rate. Knowledge of the OCE is not sufficient to determine any trends in corrosion rate for a series of experiments performed on systems having different local polarization characteristics.

Corrosion rates for pure iron in 1N HCl could be determined from cathodic polarization measurements, even in the presence of aniline, aniline derivatives and a number of alkyl-amines.[7] The efficiency of these materials as corrosion inhibitors for iron could be determined from cathodic polarization measurements. The agreement found between corrosion rates determined from polarization measurements and rates determined from the analytical determination of iron indicates that the corrosion reaction is not appreciably affected by ohmic resistance on the part of inhibitor molecules adsorbed onto the iron surface, within the inhibitor concentration range normally used.

Polar organic amines function as inhibitors of wet corrosion by forming an adsorbed layer at the metal-solution interface. Little information is available as to the relative contributions of ohmic versus activation overpotential to the inhibition process. However, the ohmic resistance contribution to the inhibition process appears to be more significant at high concentrations of inhibitor than it is at relatively low concentrations.

Aim of this investigation is to extend Stern's, Kaesche's and Hackerman's method to the study of media at pH values approaching neutrality by the use of appropriately buffered systems. This investigation also will evaluate the effect of the complex polar organic amines which are used commercially in the mitigation of corrosion of mild steel in anaerobic media in the vicinity of pH 5, both in the presence and in the absence of carbon dioxide.

Experimental Data

Materials

Acid and salt solutions were made from reagent grade materials and distilled water. The various flushing gases were passed over copper turnings heated to 700 C. This treatment was considered necessary to remove the last traces of free oxygen in these gases. The amine used as a corrosion inhibitor for iron and steel in this investigation was a product of the Armour Chemical Division and was claimed by the manufacturer to be a technical grade fatty acid amine derived from tallow and having an amine content of approximately 80 percent, calculated as diamine.

Impurities in this material were said to be other amines containing a somewhat fewer, or somewhat larger, number of methylene groups in the molecule. No further purification was attempted because this material was to be used in a study concerned with an efficiency evaluation of commercial corrosion inhibitors. Amine salts used as corrosion inhibitors in this investigation were naphthenic acid salts of the tallow amine described above.

Measurements were made with two types of electrodes: pure iron and 1020 mild steel. In each case, metal rods were imbedded in a polyester resin. The pure iron electrodes (5 mm in diameter, approximately 0.2 cm^2 cross-sectional area) were produced from sponge by powder metallurgy methods. The analytical report from the producer was as follows: 0.03 percent C, 0.01 percent O_2, 0.01 percent N_3. The mild steel rods used as electrodes were 6.5 mm in diameter (approximately 0.33 cm^2 cross-sectional area). A sample of this steel was submitted to a commercial testing laboratory; the analytical report received stated that the steel could be classified as AISI 1020 steel.

Apparatus

Test cells used were 1000 ml beakers fitted with rubber stoppers. A polytetrafluoroethylene bushing was inserted through the center of each stopper for a glass stirring paddle driven by an electric motor outside the cell. The corroding electrode, the SCE, and the platinum auxiliary electrode were mounted vertically in the cell through the rubber stopper. They were arranged with the corroding electrode at the apex of a small triangle. There was an inch between the SCE and the other two electrodes; the distance between the other two electrodes being about 1½ inches.

The glass stirring rod was located symmetrically opposite the corroding electrode. The SCE made contact with the test medium through an agar bridge. The conducting salt in the agar was potassium chloride. Test cells were main-

tained at 30 C by immersion in a constant temperature bath. The glass stirring paddle was rotated at approximately 1100 rpm, and the gases were bubbled through the test media at a rate of approximately 400 cc/min.

The potential measuring system was an electronic recorder modified to measure potentials to ± 0.25 mv between −2 and +2 volts. A regulated power supply plus a series of precision resistors provided the polarizing currents. The potential measurements were made between the corroding electrode and the SCE. Polarizing currents were impressed between the corroding electrode and the platinum auxiliary electrode. The polarizing currents were constant throughout the polarization time because resistance of the electrode system was small compared to the external ohmic resistance.

Most of the corrosion rates determined from polarization data were computed on a digital computer. An interpretive programming system was used to introduce the polarization data into the computer.

Procedure and Results

Procedure followed in obtaining potential measurements was to fill the cell with the test medium, bubble solvent-saturated and oxygen-free flushing gas through the medium, insert the corroding electrode and measure the potential at intervals until a steady state was attained, usually in approximately four hours.

In general, the steady state potentials become more negative (active) as the pH of the medium increases. Carbon dioxide in an otherwise unbuffered system resulted in a more noble steady state potential but in a buffered system the addition of carbon dioxide had no appreciable effect on the steady state potential. Addition of a second, nonconducting phase to the test medium did not have a profound effect on the steady state potential, causing only a slight shift in the noble direction. Pure iron required a longer time to achieve a steady state potential than mild steel but ultimately approached the same potential value. The rate at which steady state potentials were attained also increased with pH.

Addition of organic amines or amine salts to the system caused a shift in the steady state potential to more noble values which varied with inhibitor concentration to approximately 100 ppm. At higher concentrations the shift in potential ceased, and the steady state potential became increasingly difficult to attain. In this range, potentials were difficult to reproduce. Addition of organic amines to acid solutions has been found to cause the potential of mild steel to shift to more noble values.[16]

Cathodic Polarization Measurements in Unbuffered Systems

A series of cathodic polarization measurements were made on mild steel immersed in hydrochloric acid solutions. After the steel electrode was immersed in the test medium, periodic open-circuit potential measurements were made to ascertain that a steady state potential had been attained before any polarization measurements were made. Polarization behavior of the electrode was then checked at intervals for 12 to 18 hours following attainment of a steady state potential.

Figure 1 compares typical cathodic polarization curves obtained on mild steel in HCl solutions at pH values of 1, 2 and 5. The systems used were comparable in every other respect and show clearly the pH effect on cathodic polarization curves. Figure 2 compares helium with carbon dioxide, as a flushing gas. These curves show that the buffering action of the carbon dioxide shifted the onset of concentration polarization to a higher value of impressed current density.

TABLE 1—Variety of Conditions Used for Buffering

Condition	Electrode	Flushing Gas	Inhibitor	% Kerosene
1	Mild steel	Helium	None	0
2	Mild steel	Carbon dioxide	None	0
3	Pure iron	Carbon dioxide	None	0
4	Mild steel	Carbon dioxide	None	20
5	Mild steel	Carbon dioxide	50 ppm A	20
6	Mild steel	Carbon dioxide	x ppm B	20
7	Mild steel	Carbon dioxide	x ppm C	20

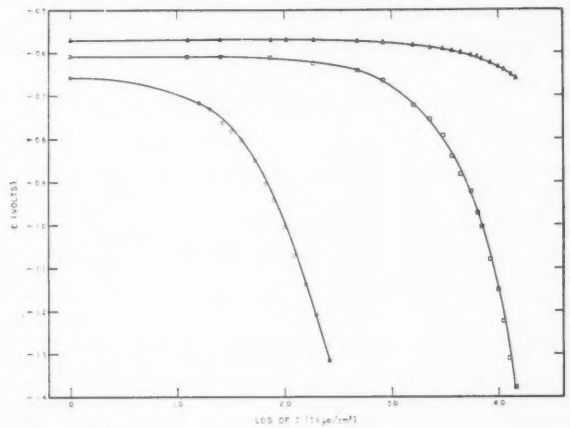
Cathodic Polarization Measurements in Buffered Systems

Cathodic polarization measurements in buffered systems were made in the same manner as those in unbuffered systems. Three buffering systems were used: (1) 0.1N acetic acid and 0.18N sodium acetate — pH 5, (2) 1.0N acetic acid and 1.8N sodium acetate — pH 5 and (3) 2.0N formic acid and 0.4N sodium formate — pH 3.

The second and third systems were used only for mild steel with oxygen-free carbon dioxide bubbled through the test solution. The first buffering system was used under a variety of conditions listed in Table 1.

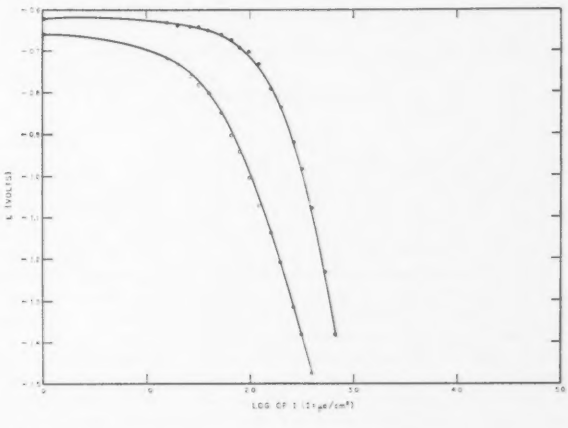
Figure 3 shows four series of measurements made on mild steel. Two were made in HCl solutions originally at a pH of 5, one using oxygen-free helium as the flushing gas, the other using oxygen-free carbon dioxide. The other two measurements were made in solutions buffered to pH 5 with 0.1N acetic acid and 0.18N sodium acetate, one using oxygen-free helium and the other oxygen-free carbon dioxide. These curves show that carbon dioxide in the test medium exerted a more pronounced effect in a system which is unbuffered than in a buffered one.

In both of the buffered systems, a Tafel relationship was observed before the onset of a significant concentration polarization contribution to the data.



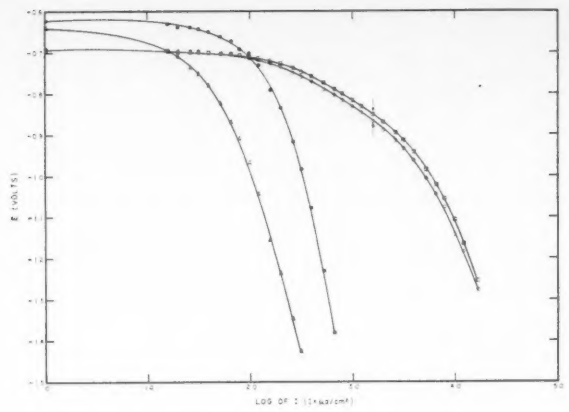
Δ pH = 1.0
□ pH = 2.0
○ pH = 5.0
MILD STEEL IN HCl

Figure 1—Effect of pH.



MILD STEEL IN HCl
○ pH = 5.0 HELIUM
● pH = 5.0 CARBON DIOXIDE

Figure 2—Effect of CO_2.



Δ pH = 5.0, HCl, CO_2

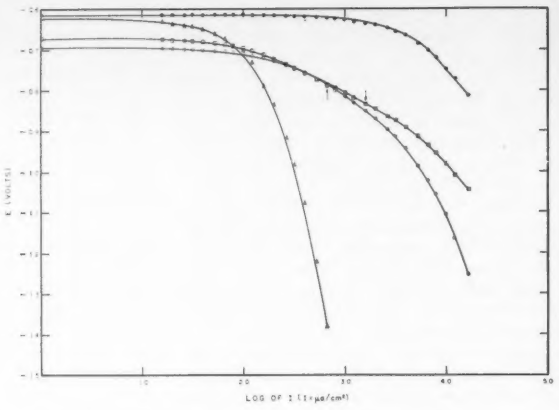
□ pH = 5.0, 1.0N HAc, 1.8N NaAc, CO_2

○ pH = 5.0, 0.1N HAc, 0.18N NaAc, CO_2

● pH = 3.0, 2.0N FORMIC ACID, 0.4N Na FORMATE, CO_2

↑ ONSET OF CONCENTRATION POLARIZATION

Figure 3—Effect of buffering and effect of CO_2.



● pH = 5.0, HCl, CO_2

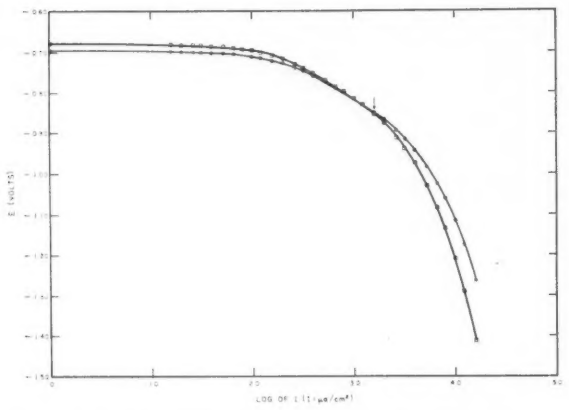
Δ pH = 5.0, HCl, He

○ pH = 5.0, HAc-NaAc, He

□ pH = 5.0, HAc-NaAc, CO_2

↑ ONSET OF CONCENTRATION POLARIZATION

Figure 4—Comparison of buffer systems.

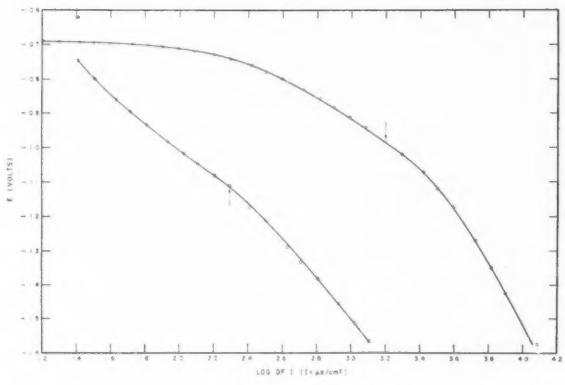


○ 0.1 N HAc – 0.18 N NaAc – MILD STEEL – CO_2

□ 0.1 N HAc – 0.18 N NaAc – MILD STEEL – CO_2
(DIFFERENT SYSTEM – 1 MONTH LATER)

↑ ONSET OF CONCENTRATION POLARIZATION

Figure 5—Effect of oil phase.



○ NO. 25e MILD STEEL

□ NO. 40a PURE Fe

↑ ONSET OF CONCENTRATION POLARIZATION

Figure 6—Mild steel vs pure iron.

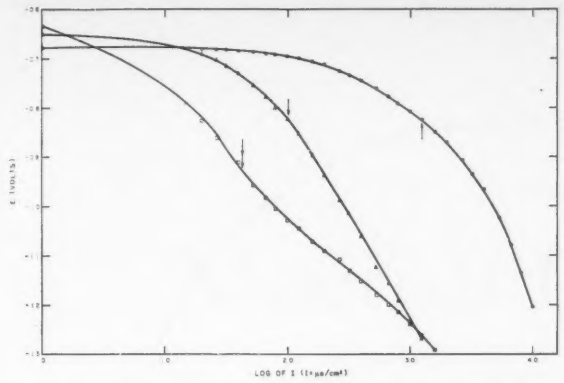
The concentration polarization effect became noticeable at approximately 1450 $\mu a/cm^2$, and the Tafel region of the curve was the straight-line portion immediately preceding this point.

Figure 4 compares the various buffering systems used. An increase in buffer capacity for a particular buffer system increased the current range over which the Tafel relationship was observed.

Figure 5 shows the effect on cathodic polarization measurements of an oil phase in the test cell. Both measurement series were made on mild steel immersed in 0.1N acetic acid and 0.18N sodium acetate, saturated with oxygen-free carbon dioxide. The systems differed only in that one contained 20 percent kerosene by volume. The data indicate that a second, non-conducting phase need not interfere seriously with the determination of corrosion rates of iron and steel from cathodic polarization measurements. However, determinations in inhibited media could be affected by the distribution of inhibitor between the two phases.

Figure 6 compares mild steel with pure iron. In both measurement series, the test medium consisted of 0.1N acetic acid and 0.18N sodium acetate, and oxygen-free carbon dioxide was bubbled continuously through the solution. For the mild steel curve, the concentration polarization effect started at a log i value of approximately 3.2, and the Tafel region was the straight-line portion immediately preceding this. For the pure iron curve, the concentration polarization effect started at a log i value of approximately 2.3, and again the Tafel region precedes this. The lower corrosion rate of pure iron is seen in these curves.

Figures 7 and 8 illustrate the effect on cathodic polarization measurements of various concentrations of inhibiting materials used in the petroleum industry. Figure 7 shows a dependence of corrosion rate on Inhibitor B concentration to approximately 100 ppm. At this concentration, higher values of impressed current yield data which are obscured by a resistance polarization con-



○ BUFFER, KEROSENE, pH 5.0, CO_2, BLANK

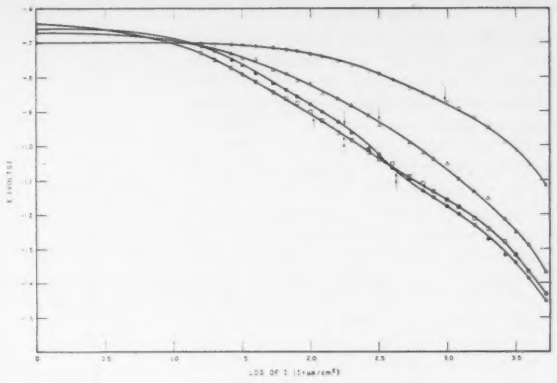
△ BUFFER, KEROSENE, pH 5.0, CO_2, 50ppm INHIBITOR B

□ BUFFER, KEROSENE, pH 5.0, CO_2, 100 ppm INHIBITOR B

↑ ONSET OF CONCENTRATION POLARIZATION

↓ ONSET OF RESISTANCE POLARIZATION

Figure 7—Effect of inhibitors with concentration to about 100 ppm.



○ BLANK

△ 20ppm INHIBITOR C

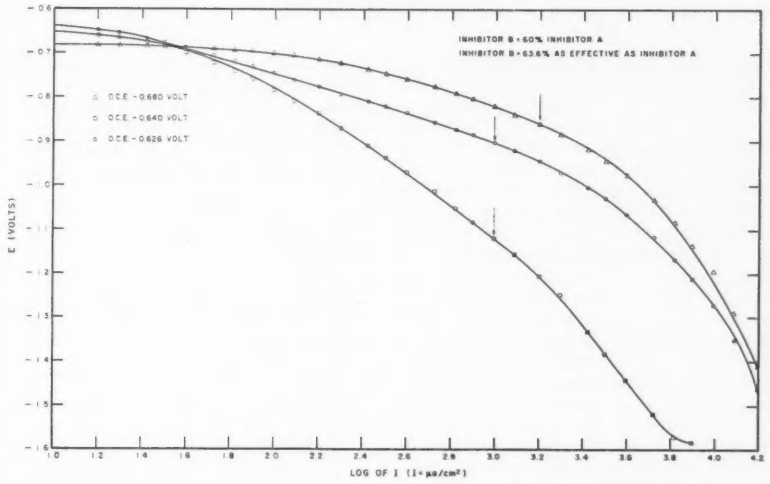
● 40ppm INHIBITOR C

□ 60ppm INHIBITOR C

↑ ONSET OF CONCENTRATION POLARIZATION

↓ ONSET OF RESISTANCE POLARIZATION

Figure 8—Effect of inhibitors to concentration of 60 ppm.


INHIBITOR B + 60% INHIBITOR A
INHIBITOR B + 63.6% AS EFFECTIVE AS INHIBITOR A
O.C.E. - 0.680 VOLT
O.C.E. - 0.640 VOLT
O.C.E. - 0.626 VOLT
E (VOLTS)
LOG OF I (I = μa/cm²)

△ NO. 32b - 80% 0.1 N HAc + 0.18 N NaAc - 20% KEROSENE, CO_2 SATURATED, BLANK - 508 MINUTES - 83.75 μa/cm²

□ NO. 33b - 80% 0.1 N HAc + 0.18 N NaAc - 20% KEROSENE, CO_2 SATURATED, 50ppm INHIBITOR B - 411 MINUTES - 40.74 μa/cm² (51.4% EFFECTIVE)

○ NO. 41a - 80% 0.1 N HAc + 0.18 N NaAc - 20% KEROSENE, CO_2 SATURATED, 50ppm INHIBITOR A - 438 MINUTES - 16.03 μa/cm² (80.9% EFFECTIVE)

↑ ONSET OF CONCENTRATION POLARIZATION

Figure 9—Amine vs amine salt.

tribution and by a contribution from concentration polarization. Above this concentration, an increase in inhibitor concentration appears to show no further effect on the activation polarization.

The resistance polarization contribution becomes noticeable at a log i value of approximately 1.6; it becomes increasingly more pronounced as it overshadows the concentration polarization contribution more and more. The Tafel region is taken as the straight-line portion of the curve immediately preceding the onset of resistance polarization. Figure 8 shows a dependence of corrosion rate on Inhibitor C concentration to the highest concentration used—60 ppm.

At this concentration, the effect of concentration polarization becomes noticeable at a log i value of approximately 2.0; that of resistance polarization at a log i value of approximately 2.2. The Tafel region is taken as the straight-line portion of the curve immediately preceding the onset of concentration polarization. Inhibitor C is more effective than Inhibitor B under the test conditions employed. Both were tested under identical conditions in systems which included oxygen-free carbon dioxide, mild steel, kerosene, and the acetate buffer solution in proportions calculated to yield an initial pH of 5.

Figure 9 presents data which compare the effects of a technical grade fatty acid amine at a concentration of 50 ppm with the naphthenic acid salt of this amine at the same concentration. The amine is referred to as inhibitor A, and the amine salt is Inhibitor B. Inhibitor B consists of approximately 60 percent Inhibitor A. The data indicate that Inhibitor B is approximately 60 percent as effective as Inhibitor A. This is construed as evidence that, when an amine salt is used as a corrosion inhibitor, inhibition is provided by the amine portion of that salt.

Figure 10 shows two series of measurements made on different but identical systems to indicate the degree of reproducibility obtained in these measurements. Both were made on mild steel in a system buffered with 0.1N acetic acid and 0.18N sodium acetate to a pH of 5, with carbon dioxide bubbled continuously through the test medium. These data were included to show that the irreproducibility commonly associated with polarization measurements in complex systems can be avoided.

Discussion

Because concentration polarization effects constitute the only significant deterrent to extending the use of this method to the determination of corrosion rates of iron and steel in media at higher pH values, the use of buffers of adequate capacity is suggested as a means of eliminating this deterrent.

The use of a buffer provides a range in the activation polarization curve that is relatively unaffected by concentration polarization and which can be extrapolated back to the steady state open-circuit potential to yield a corrosion rate in terms of current density. This effect is apparently not peculiar to a particular buffer system because it was demonstrated with more than one buffer system.

Because it had previously been demonstrated that the use of a buffering system consisting of acetic acid and sodium acetate does not result in resistance polarization because of adsorption, and because the other buffer systems used in this investigation produced results very similar to those obtained with acetic acid and sodium acetate, it is concluded that none of the buffering ions used are adsorbed onto the test electrodes.

Presence of a second, non-conducting phase did not exert any appreciable effect on the determination of corrosion rates by this method. If the two phases are kept dispersed by adequate agitation of the test medium and if in this dispersion the conducting phase is continuous, no great effect should be expected on the polarization measurements.

The difficulty experienced in achieving steady-state potentials in the presence of amine inhibitors at higher concentrations can be attributed to the less stable ohmic resistance polarization contribution of those inhibitor molecules which are not directly adsorbed onto the metal surface. The inhibitor monolayer adsorbed directly onto the metal surface is probably held, at least in part, by chemisorption forces; those molecules which are not in direct contact with the metal surface are held by weaker physical adsorption forces.

At relatively high inhibitor concentrations, resistance of the adsorbed inhibitor is seen to fluctuate erratically as though relatively large agglomerations of inhibitor molecules are separated from the adsorbed layers and subsequently readsorbed. The existence of two types of adsorption forces is also suggested by the fact that the ohmic resistance of an inhibitor film can be made to disappear by simply rinsing the test electrode with fresh solvent; repeated rinsings do not affect the shift in steady state potential observed upon the addition of an amine inhibitor to a corroding system.

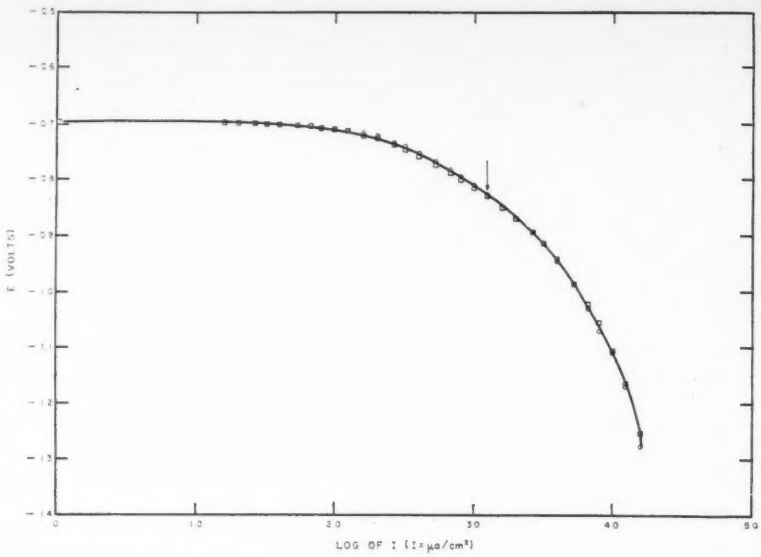


Figure 10—Data reproducibility.

The method of Kaesche and Hackerman for determining the efficiency of pure organic amines as corrosion inhibitors of iron has been extended to include more complex carboxylic acid salts of technical grade polyamines in the forms used commercially for control of iron corrosion. The carboxylic acid portion of the amine salts used contributes little to the actual process of inhibition although various advantages may be derived from the use of the salt in preference to the free amine such as increased solubility in the corrosive medium.

Efficiency of the complex materials used commercially for corrosion inhibition of iron and steel can be determined in the presence of carbon dioxide, in the presence of a second, non-conducting phase and in pH ranges approaching neutrality. The method described here provides a rapid and relatively accurate means of evaluating the efficiency of corrosion inhibitors in systems which are in pH ranges of commercial interest.

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Any discussion of this article not published above will appear in the December, 1959 issue.

Publication 59-18

Some Fundamental Aspects of

Materials Deterioration in the Atmosphere

Four educational lectures presented at the 14th Annual Conference of the National Association of Corrosion Engineers at San Francisco, March 17-21, 1958.

CONTENTS

Mechanisms of Atmospheric Corrosion of Ferrous Metals by C. P. Larrabee

Mechanism by Which Non-Ferrous Metals Corrode in the Atmosphere by P. M. Aziz and H. P. Godard

Principles and Procedures Employed in Design and Interpretation of Atmospheric Corrosion Tests by H. R. Copson

Deterioration of Materials In Polluted Atmospheres by J. E. Yocum

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FOREWORD

In March, 1958 at the National Association of Corrosion Engineers' meeting in San Francisco, a series of educational lectures on atmospheric corrosion and deterioration was presented by outstanding specialists. While there has been great progress in the past 50 years in this field, there still is a tremendous annual economic loss because of attack by the elements on externally exposed installations of all kinds. Much research and development still is needed to find less expensive ways of protecting metals and other materials from atmospheric damage.

This series of carefully prepared lectures, giving the status-of-the-art, is intended as a convenient reference to corrosion engineers and investigators. They also should serve as a stimulus to students of corrosion, some of whom may be alerted to the need for specialization in this field.

F. W. FINK

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Aziz

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Mechanisms by Which Ferrous Metals Corrode In the Atmosphere*

By C. P. LARRABEE*

Abstract

The electrochemical theory of corrosion, by which the corrosion of steels, in solutions is readily explained, also is used to explain the start of atmospheric corrosion on ferrous surfaces. Effect of contaminants both in the atmosphere and on the steel surface is described. Experiments leading to this conclusion are illustrated.

The part played by various alloying elements in retarding atmospheric corrosion, once a rust film is formed, is discussed. The relative effects of heavy and light deposits of sea salt on ferrous specimens are shown. 4.2.1

Introduction

MAN OWES his progress in science to his persistence in finding the answers to the "whys" and "hows" that confront him. In the past, it has been the custom of some to ascribe to the supernatural everything not subject to simple explanation. Pliny attributed the tendency of iron to rust to a curse inflicted by the gods on that metal because of its use in war. Since the general acceptance of the electrochemical theory of corrosion (to be discussed later herein), there has been more progress in understanding corrosion and what to do about it than there was in the preceding 19 centuries.

Probably because of the relative ease by which experimenters could control the conditions, much of the fundamental work on the "whys" and "hows" of corrosion has been done in simple solutions. As will be discussed later, so many variables affect corrosion in the atmosphere that controlled experiments are difficult. Although Davy in 1800[1] theorized that the action of a galvanic battery was due to electrochemical action, it was not until 1856 that de La Rive[2] claimed that the atmospheric corrosion of iron also was electrochemical in nature. Because of inadequate communications existing at the time, these ideas were not generally known or remembered after a few decades. Indeed, in 1903, when Whitney[3] published his "original" ideas, he received credit for a "new" theory.

Why Iron Corrodes

Before going into detail on the "hows" of the corrosion of ferrous metals, examine briefly the "why." Simply stated, iron has a natural tendency to corrode because there is a large negative free energy difference for the reaction of iron to form iron oxide. In other words, the natural process by which iron returns to the same chemical composition as its ores is sometimes termed "corrosion." Gold, platinum, and the other so-called "noble metals" that do not corrode in the air are found chemically uncombined in nature.

How Iron Corrodes

Previous to the general acceptance of the electrochemical theory, there were five theories that had been advanced to explain the corrosion of metals. There were only two that had adherents at the turn of this century, and these were not abandoned until some years afterwards. Even these latter two can be dismissed now after a few words of explanation.

Hydrogen Peroxide Theory

According to Wilson[4] this theory assumes that when iron, water and oxygen are in contact, ferrous oxide and hydrogen peroxide are formed. Half of the hydrogen peroxide reacts with the ferrous oxide to form rust, which is an insoluble basic oxide. The other half of the peroxide is supposed to react with more iron, thus making the reaction self-perpetuating. Slight traces of hydrogen peroxide are found during the slow oxidation of certain metals, but none has been found in the oxidation of iron. Another fact that seemed to lend credence to this story was the inhibiting effect of chromates on the rusting of iron when immersed in water. Chromic acid destroys hydrogen peroxide.

This evidence is offset by the fact that several oxidizing agents which destroy the peroxide do not inhibit the rusting of iron. The theory has been discarded because of its inability to explain satisfactorily many observed facts about corrosion.

Carbonic Acid Theory

This theory, which had more adherents than the hydrogen peroxide theory, postulates, according to Wilson,[4] that rusting is always started by an acid, even a weak one such as carbonic acid. Iron is assumed to be converted to the ferrous salt, which is oxidized to ferric hydroxide, an insoluble precipitate. However, the carbonic acid theory cannot explain the following experiment.[4] Iron is immersed in water which has been freed of oxygen and carbon dioxide. According to experiments described by Cushman and Gardner[5] no rusting occurs, but when oxygen from which carbon dioxide has been completely removed is introduced, rust appears in a few moments. When oxygen-free carbon dioxide is introduced into a similar flask, no rusting occurs, showing the necessity for the presence of oxygen. Rusting of iron also occurs in weak alkaline solutions, which is another point against this theory.

Electrochemical Theory

Briefly, this theory is based upon the setting up of an electric cell by one of several processes. If an electric current results, metal goes into solution at one pole (anode) and hydrogen is deposited at the other pole (cathode). Because it is considerably harder to control the variations in water films such as those caused by rain on sloping surfaces or droplets such as deposited by dew, most of the experimental work on the electrochemistry of corrosion has been done with solutions, usually containing some dissolved salts to increase their conductivity. However, because the subject of this article is the mechanism of the atmospheric corrosion of iron, those factors affecting the corrosion of unrusted iron and their relationship to the electrochemical theory will be discussed.

According to the electrochemical theory, there is a flow of electric current when corrosion occurs. For an electric current to flow, there must be an electrolyte on the surface of the metal. The electrolyte can be water in the form of a film from rain, droplets from dew, or as an absorbed film on a foreign particle. In most cases of atmospheric corrosion, distances between anodes and the cathodes are relatively small compared to distances between these active sites in liquids.

The difference in potential between an anode and a cathode is the amount of driving force of the corrosive action. It is theorized that an iron atom carrying two positive electrical charges detaches itself from the metal surface and enters the solution as an ion. The two positive charges reach the cathode either on the ferrous ion or on two of the millions of available hydrogen ions. The negative charges (electrons), corresponding to the positive charges on the ferrous ions, travel through the metallic part of the circuit to the cathode where they meet the hydrogen ions in the solution. When the two charges are neutralized the two hydrogen atoms may combine as a gas, or in the case of atmospheric corrosion, they usually will combine with oxygen to form water.

The ferrous ions unite with the hydroxyl ions to form ferrous hydroxide. In the presence of air, this soon becomes hydrated ferric oxide, the familiar rust.

The previously described electrical cell consists of a metallically-connected anode and cathode immersed in a liquid. The amount of current that flows is determined by the open circuit potential difference between the anode and cathode, the amount of electrochemical polarization occurring at anode and cathode, and the electrical resistance of the solution. When unrusted iron is dry, external resistence between the anode and cathode areas is infinite, so no current can flow, and therefore corrosion cannot occur. All are familiar with the fact that iron does not

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rust as long as it is clean and dry. The necessity of using the word "clean" will be discussed later.

Invisible Oxide Films

When iron remains dry for an appreciable time, there is formed on its surface an invisible film containing oxygen.

Mears and Evans[6] demonstrated experimentally that the amount of protection given by the air-developed invisible film on iron was dependent upon many factors. Their work was done with droplets of water so small that the conditions may have approached those existing in the atmosphere.

Vernon,[7, 8, 9] by controlled atmospheric tests, showed that the air-developed film protected iron from corrosion when in air at a relative humidity of 99 percent. If, however, the film was exposed to nearly moisture-saturated air having 0.01 percent or more of sulfur dioxide, corrosion occurred. Also, if the iron were first exposed to air containing sulfur dioxide and subsequently exposed to air at 80 percent relative humidity, corrosion also would occur. Evidently, some change in the structure of the invisible oxide film is caused by exposure to sulfur dioxide. It is probable that adsorbed water vapor on salts deposited from the air produces the same reaction. Corrosion has been observed under a microscope to spread from several tiny spots where certain salts have been placed until finally the whole surface is covered with visible rust.

Skorchelletti and Tukachinsky[10] have extended Vernon's pioneering work under carefully controlled conditions. They have further studied the conditions that are necessary to break down the "passive" film and to form the first trace of rust. As seen in Figure 1 (Figure 4 of Skorchelletti and Tukachinsky paper) the amount of SO_2 in the air played a large role in the amount of corrosion of clean steel exposed at 100 percent relative humidity. They measured corrosion by having the specimen hung on one arm of a microbalance having a sensitivity of 3×10^{-6} g per division. The specimen was surrounded by the atmosphere being studied and measurements of increase in weight were made in situ. By this method, they report that the critical humidity (sufficient to cause corrosion as measured by an increase in weight) was about 85 percent when 0.02 percent SO_2 was present. This is only about 7 to 10 percent higher than Vernon's comparable value.

The use of the balance did not permit an exact determination of the critical humidity in the absence of traces of SO_2. However, they did ascertain that once a specimen had been exposed to more than 0.02 percent SO_2, even at humidities less than the critical, heating in a vacuum to temperatures of 626 F did not give the oxide film its original resistance to humidity.

Tagaya and Isa[11] have studied the rusting of polished steel by viewing the process with a microscope. Four types of rust were observed as shown in Table 1.

Stainless Steels

Invisible oxide films that render chromium steels "stainless" in chloride-free atmospheres have been isolated by ingenious methods. A few seconds exposure to air is sufficient time for the film to be reformed after its removal by chemical means. The excellence of this film in protecting the steel from attack by an industrial atmosphere is shown in Figure 2. This shows the surface of a 17 percent chromium steel after an exposure of 20 years. The lower half of the specimen was dipped in hot caustic soda solution which removed deposited dirt but would not have removed "rust" if any had been present.

The effect of chlorides in breaking down the oxide film is illustrated in Figure 3. Here is shown the same type of steel (17 percent Cr) after a 3-month exposure in a chloride-contaminated industrial atmosphere. That the concentration of chlorides plays a vital part in the staining of chromium steels is illustrated in Figure 4. It is seen that a 28 percent chromium steel remained spotless when exposed for three years at 800 feet from the seashore but stained badly when exposed 80 feet from the shore.

Progress of Corrosion

Of great practical interest is the question "Does rusting, once started, proceed at a uniform rate?" Fortunately, the answer is "no." The all too familiar reddish-brown rust is a mixture of hydrated iron oxides. Depending upon the amount of pollution in the atmosphere and the composition of the steel, this oxide film is more or less protective. Of all the common pollutants, sodium chloride, typified by sea salt, is the greatest destroyer of the protectiveness of the rust film on carbon steel. Oxides of sulfur are a poor second and when both pollutants are nearly absent the atmospheric

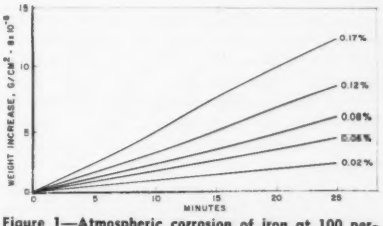

Figure 1—Atmospheric corrosion of iron at 100 percent relative humidity with increasing SO_2 concentrations. Data from Skorchelletti and Tukachinsky.[10]

Figure 2—Type 430 (17 Cr) steel after 20 years in industrial atmosphere. Lower half cleaned in caustic solution.

TABLE 1—Types of Rust on Polished Steel
(Data of Tagaya & Isa)

A	Dotted (smallest particles)
B	Granular (Grow)
C	Thread-like
D	Lumpy—Sources: 1) Cl & SO_4 particles; 2) Few of the dew drops; 3) Fe_2O_3 particles

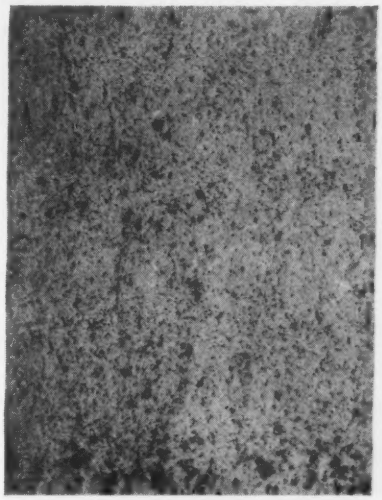

Figure 3—Type 430 (17 Cr) steel after three months in industrial atmosphere contaminated with chlorides. Scale: ¼ actual size.

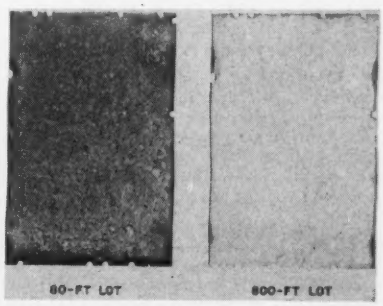

Figure 4—Type 446 (28 Cr) steel after 3½-year exposure at 80 and 800 feet from ocean.

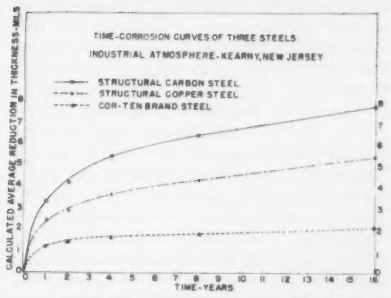

Figure 5—Time-corrosion curves of three steels in industrial atmosphere, Kearny, N. J.

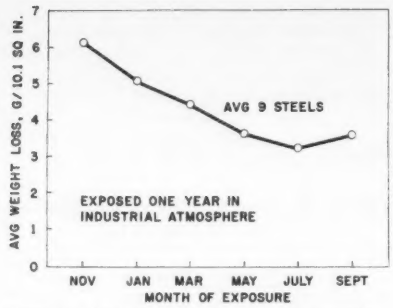
AVG WEIGHT LOSS, G/10.1 SQ IN.
AVG 9 STEELS
EXPOSED ONE YEAR IN INDUSTRIAL ATMOSPHERE
NOV JAN MAR MAY JULY SEPT
MONTH OF EXPOSURE

Figure 6—Effect of month of exposure on corrosion of steel.[12]

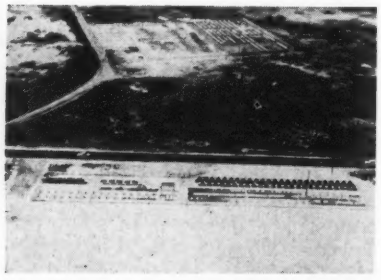

Figure 7—Test lots at Kure Beach, N. C. average 80 feet and 800 feet from surf line.

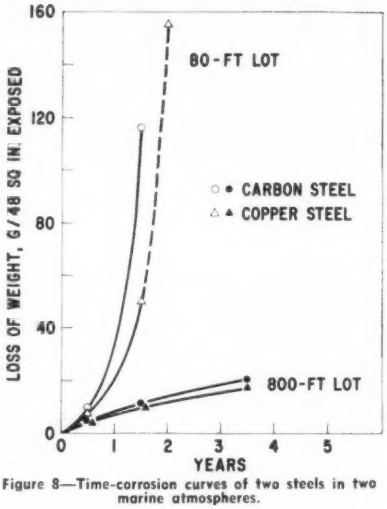

Figure 8—Time-corrosion curves of two steels in two marine atmospheres.

corrosion of steel is not significant. It is true that at locations where dew is deposited on steel, rusting as evidenced by discoloration soon takes place, but in the absence of chlorides or sulfates this rust film quickly assumes a protective nature and corrosion extends for only a few ten-thousandths of an inch into the surface.

As seen in Figure 5, long-time atmospheric tests have shown that after a comparatively few years attack in an industrial atmosphere is essentially linear with time. Attention is called to the nearly straight time-corrosion curves after the initial rust film was stabilized. In Table 2 are shown the calculated average corrosion rates for the most resistant steel during the various exposure periods. After eight years the average corrosion rate is

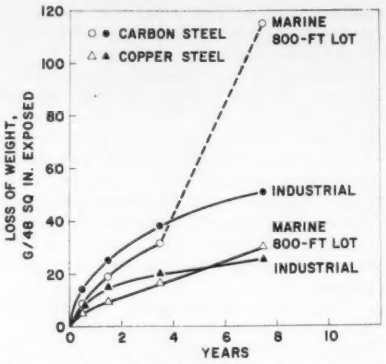

Figure 9—Comparison of corrosivity of marine and industrial atmospheres.

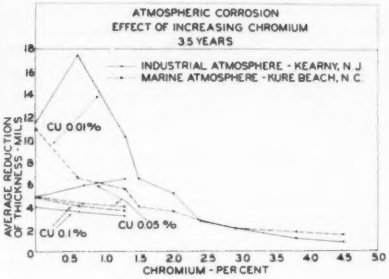

Figure 10—Effect of minor alloy (Cr and Cu) additions on atmospheric corrosion. 3.5 years.

very low: 0.008 mil per year. Studies of atmospheric corrosion in industrial atmospheres show the effects of changes in weather. Schramm and Taylerson[12] published the data shown in Figure 6 showing the results of tests started at two-month intervals from November to September and exposed for one year.

During the last 18 years extensive testing of steels in marine atmosphere has taken place at Kure Beach, North Carolina. A view of the two test lots is shown in Figure 7. The one in the foreground is designated as "80-foot lot" and the larger one as "800-foot lot." That proximity to a source of chloride contamination makes a great difference in the corrosion rate of carbon and copper steel is seen in Figure 8. Unquestionably, it is the accelerating effect of the sea salt on the corrosion of the groundward exposed surfaces that causes the difference. The concentration of salt on the skyward surface is low because of the leaching action of rain. There is much more spray at the 80-foot lot, hence much more corrosion.

The data in Figure 9 (Marine and Industrial) show that sometime between 3.5 years and 7.5 years the build-up of sea salt on the groundward surface of a carbon steel specimen at the 800-foot lot caused a major increase in the corrosion rate. The copper steel did not show this effect after 7.5 years but the time-corrosion curves of both steels in marine atmospheres cross those of the same steels in industrial atmospheres.

Why are Certain Rust Films Protective?

The "why" of this phenomenon is not easily apparent. Copson[13] has postulated that with low alloy steels containing nickel, copper, or chromium, the sulfates of these elements are formed during the corrosion process. Because the sulfates of these elements are more insoluble than the sulfates of iron, the pores in the rust on low alloy steel become clogged and prevent access of water to the steel itself. This theory, while serving in some cases, is too simple to explain the many effects of various combinations of alloying elements some of which, like phosphorus and silicon, do not form sulfates.

Data in Figure 10 give an indication of how complex this problem is. When corrosion varies as shown here with only minor changes in steel composition, it is evident that a simple explanation will not suffice.

On the basis that simple systems are more easily studied, let us consider the effect of the addition of copper to steel. In 1919, Buck[14] published the fact that steels containing over 0.15 percent copper had appreciably more atmospheric corrosion resistance than steels with less than 0.05 percent copper. Buck believed that the copper in the steel combined with the sulfur in the steel to form copper sulfide and that this was not so deleterious corrosion-wise as the otherwise present ferrous sulfide.

This was later shown to be true by the data in Table 3. In this experiment, two irons, one having 0.0004 percent Cu, the other 0.2 percent (A and B in Table 3), and each having 0.03 percent sulfur were used. Both were annealed at 2400 F for one week in dry H_2. This treatment reduced the sulfur to less than 0.001 percent but did not affect the copper content. Specimens of the two non-annealed and the two annealed irons were exposed to the atmosphere for 1.5 years. The atmospheric corrosion losses of three low-sulfur irons were equal, but, the loss of the specimen containing sulfur but no copper was five times as great. Thus, when there was no sulfur in the steel, the pres-

TABLE 2—Calculated Average Corrosion Rates Low Alloy Steels in Industrial Atmosphere

Year	Skyward Surface Mils Per Year	Ratio*
1st	0.5	60
2nd	0.06	8
3rd & 4th	0.045	6
5th, 6th, 7th, & 8th	0.025	3
9th to 16th	0.008	1

* Of the loss per year to the annual loss during the 9th through 16th years.

TABLE 3—Effect of Sulfur and Copper in Iron on Atmospheric Corrosion

Iron	Per Cent S	Per Cent Cu	Corrosion[1]
A	0.03	0.0004	5
B	0.03	0.2	1
C (A annealed)[2]	0.001	0.0004	1
D (B annealed)[2]	0.001	0.2	1

[1] Based on weight loss after 1.5 years.
[2] 0.064-inch-thick specimens annealed at 2400 F in dry H_2 for one week.

ence of copper did not improve corrosion resistance.

Recent laboratory work by Skorchelletti and Tukachinsky[10] indicates that the colloid-chemical properties of the rust appear to be the major agent in the formation of a film with measurable electrical conductivity. Without such a conducting film, further corrosion is impossible. They also reported that for the rust on a steel of a certain composition, there was a critical humidity above which rusting proceeded even without liquid water being present. With some alloy steels, the critical humidity was so high that corrosion occurred only when the steel was actually wet. Differences in the electrical resistance of rust films on various steels were found. With high resistances, the current necessary for solution of appreciable iron could not flow. This type of investigation promises a fruitful field for future work on the rusting of alloy steels.

Conclusion

All investigators are now in general agreement on the electrochemical theory for the initial rusting of steel in atmosphere. However, once the first rust has formed, the complexities of the system are so great that much work remains to be done before there can be developed a full understanding of the "whys" and "hows" of the rate of the continuing rusting process.

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Mechanism by Which Non-Ferrous Metals Corrode in the Atmosphere*

*By P. M. AZIZ and H. P. GODARD**

Abstract

Literature dealing with the mechanism by which non-ferrous metals corrode in the atmosphere is reviewed, particular emphasis being given to the important series of papers published by W. H. J. Vernon and his co-workers in the period extending from 1923 to the present. The influence of the common atmospheric constituents is discussed and related to the chemical composition and properties of the corrosion produced film formed. The influence of these on the atmospheric corrosion rate is also discussed in relation to the nature of the corrosive atmosphere. The atmospheric corrosion of nickel, copper, zinc, aluminum and magnesium is discussed.

4.2.1

Introduction

WHILE a great deal of work dealing with the atmospheric corrosion of non-ferrous metals has been carried out, the aim of most of this has been the accumulation of numerical data on the rates of attack of representative metals under a variety of exposure conditions. Little of this work has had as its aim the basic understanding of the mechanism whereby these metals undergo attack in the atmosphere. The most important work dealing with this subject has been carried out under the sponsorship of the Atmospheric Corrosion Research Committee of the British Non-Ferrous Metals Research Association with Drs. W. H. J. Vernon and J. C. Hudson as principal investigators. This review refers extensively to their work[1-8] and the importance of many of the fundamental principles established by them is emphasized.

A. General Principles

1. Controlling and Promoting Factors

An insight into the nature of the processes occurring during atmospheric corrosion can be best obtained by a comparison with the corrosion process taking place under conditions of total immersion. In any chemical reaction which consists of a series of consecutive steps, the rate at which the overall change can take place is determined by the rate of the slowest step which may be termed the controlling factor in the reaction. Vernon[4] has pointed out that immersed and atmospheric corrosion differ, not in the type of reaction that occurs but in their respective controlling factors. Thus at first sight it might appear that the presence or absence of water is the distinguishing feature and that atmospheric corrosion might be considered a gas-metal heterogeneous reaction and immersed corrosion a metal-liquid reaction. However, since most cases of atmospheric corrosion occur in the presence of water vapor, which results in films of adsorbed moisture, or in the presence of rain, it can be seen that this is not the case.

In the common case of immersed corrosion under cathodic control the rate of the reaction is often controlled by the diffusion of oxygen through the solution to the local cathode areas. In the case of the acid dissolution of a metal the controlling factor is generally the hydrogen overvoltage on the metal or less often the rate of diffusion of the metal cation away from the local anodes. In the case of atmospheric corrosion these processes clearly can not be controlling since, for example, the access of oxygen to the surface will be virtually unlimited. Rather, the controlling factors under these conditions are the relative humidity and the presence in the atmosphere of pollutants such as hydrogen sulfide or sulfur dioxide. These latter play a very decisive role in the process which is out of all proportion to their concentration. The reaction rate in general depends less on their concentration than on their simply being present and in fact they are effective in very small concentrations.

It should be noted, however, that Hudson and Stanners[9] have found that the rate of atmospheric corrosion of zinc is roughly proportional to the sulfur pollution. Vernon has distinguished between the effect of these pollutants and the atmospheric water vapor by terming the former "promoting" factors, since they initiate reaction, and restricts the term "controlling" factor to such items as water vapor which influence the actual rate of reaction. Another controlling factor of great importance, the corrosion product film, is produced by the reaction itself. If this is coherent and protective in nature it will limit the access of the corrosive environment to the metal surface and the reaction rate will depend on the film thickness in much the same fashion that the rate of reaction of a metal surface with pure, dry oxygen is limited by the film of surface oxide.

These principles can be illustrated by the following examples:

(a) **Sulfides as Promoting Factors.** If copper is exposed to an atmosphere containing traces of hydrogen sulfide it rapidly develops the characteristic and well known tarnish film. While this film thickens by a parabolic law similar to that followed by the oxidation of copper in pure, dry air the rate of reaction is very much greater in the presence of sulfide. In

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* Aluminium Laboratories, Ltd., Kingston, Ont., Canada.

addition, the rate of tarnishing is proportional to the sulfide content of the atmosphere[1,2]. Analysis of the surface film has shown that it consists of a mixture of cuprous sulfide and cuprous oxide with the latter predominating. In addition, the presence of water vapor does not increase the reaction rate and, in fact, at sufficiently low sulfide concentrations, will inhibit the reaction[2]. Clearly then in this reaction hydrogen sulfide is the promoting factor while the film thickness is the controlling factor.

One additional fact should be mentioned here since it illustrates a general principle applicable to atmospheric corrosion which will be discussed in greater detail later. If the copper is first exposed to pure air so as to permit the development of a thin film of oxide, it will resist tarnishing when exposed subsequently to a sulfide containing atmosphere. This is an example of a principle first discovered and demonstrated by Vernon[2] and Hudson[3] that the rate of attack is governed by the atmospheric conditions prevailing at the time of first exposure of the sample.

(b) **Principle of Critical Humidity.** As a further example, the behavior of copper exposed to air contaminated with sulfur dioxide and containing water vapor can be considered. In the complete absence of water vapor sulphur dioxide has no influence on the normal room temperature oxidation that takes place in dry, pure air. In the presence of water vapor, however, the rate of attack increases and depends on both the relative humidity and the sulfur dioxide concentration. This behavior is shown in Figure 1[10]. The ordinates are the weight increments observed after an exposure period of about 30 days. The weight increment time curves established by Vernon showed an initial rapid increase for the first few days and then rapidly flattened toward the time axis and were essentially parallel to this axis after about thirty days' exposure.

Two striking facts are brought out by these results. The first is the minimum observed at a sulfur dioxide concentration of about one percent and the second is the sudden increase in rate at relative humidities greater than 63 percent. The minimum is related to the composition of the corrosion product film. At one percent the film is normal copper sulfate, below this concentration it is the basic sulfate, while above it is the acid salt. The relevance that this observation has for the atmospheric corrosion of copper under conditions of normal exposure will be discussed later. The second fact relates to a general principle developed by Vernon[2] and Hudson[3] concerning the influence of humidity on atmospheric corrosion rates, the principle of critical humidity. This may be stated as follows: In a corrosive humid atmosphere a critical humidity exists below which the corrosion rate is very slow but above which the rate increases discontinuously to a much greater value. It has been shown[3] that this phenomenon is related to the hygroscopicity of the corrosion product film and the critical humidity is that humidity at which it becomes capable of absorbing water vapor.

Thus, in this case, the water vapor content of the atmosphere can be considered the controlling factor while the sulfur dioxide can be considered the promoting factor.

The important role played by sulfur dioxide in the atmospheric corrosion of metals is further brought out by the work of Schikorr[11,12,13] and Hudson and Stanners[9]. Schikorr measured the total amount of corrosion occurring on a series of metals exposed to the open atmosphere together with the quantity of sulfur associated with the corrosion product on each. As control a sample of marble was also exposed. The results are presented in Table 1.

The values for metal and sulfur in the corrosion product range between 8 and 80 millimols per square meter of surface. It is remarkable that the metal equivalents corroded are practically the same for metals as different as lead, zinc, copper, brass and nickel, and are similar to the amount of marble deteriorated. No less surprising is the fact that the amounts of sulfur involved are quite similar, again in view of the widely differing characteristics of the metals.

The amount of sulfur is equivalent to 50-70 percent of the metal corroded, for which a probable reaction is postulated as follows:

$$2Me + 2H_2SO_3 + O_2 = 2MeSO_4 + 2H_2O$$

The fact that more metal was consumed than can be accounted for by this equation can be explained by the formation of basic sulfates. It is evident that in spite of differences in the chemical and electrochemical characteristics of the different metals, the initial rate of corrosion was determined mostly by the quantity of sulfur that came in contact with the metal surface, which also determined the rate of reaction with the non-metallic marble.

Hudson and Stanners[9] found that the rate of atmospheric corrosion of zinc was roughly proportional to the extent of sulfur pollution for a series of panels exposed for one year at sixteen sites in Great Britain.

The exact mechanism whereby the sulfur attacks the metal surface is not clear in every case. Oxidation to the trioxide with absorption of atmospheric water vapor to produce sulfuric acid, either by reaction with adsorbed water or with rain water, may occur with subsequent direct attack of the metal to form the sulfate which will then hydrolyze to the basic sulfate. On the other hand, the sulfur dioxide may dissolve in a surface film to form sulfurous acid and then the metal sulfite, which by reaction with oxygen, will go to the sulfate. In a few cases the mechanism is known and these will be discussed under the appropriate metal.

2. *Influence of Rain and General Atmospheric Conditions*

In addition to dependence on the factors already discussed, the severity of atmospheric corrosion or the corrosivity of an atmosphere will in general vary with locale and from one time period to another. In addition to the relative humidity and the extent of industrial pollution, the amount and frequency of rainfall, the amount and direction of the wind, the amount of fog, the proximity to the sea, the amount of sun, and the average temperature all will have some bearing.

The effect of rain is complex. It can increase the corrosion rate of some metals while it decreases that of others. Thus, in the case of nickel, bold exposure to the elements will decrease the corrosion rate compared to that observed for a sheltered location.[3] This example will be discussed in greater detail later. Rain, washing the surface of a specimen, can carry away corrosive atmospheric pollutants on the surface, thus reducing the corrosion rate; on the other hand it can dissolve and carry away any soluble corrosion products which may be giving some protection. Fogs and dews, in general, will be deleterious, because they have no washing effect on the surface but cause the formation of surface films of moisture which can absorb pollutants from the atmosphere.

Wind, sun and temperature can play an important role in atmospheric corrosion because they will determine the rate of drying and the length of time that the surface remains wet. In this respect Cartwright[14] and Hudson[15] have pointed out average values of the temperature and humidity do not adequately characterize a corrosive atmosphere but rather the variation in these quantities throughout the exposure period should be considered.

It is difficult to generalize about the relative corrosiveness of marine and industrial atmospheres. In general, it can be said that the industrial will be more aggressive than the marine, although this

TABLE 1—Atmospheric Corrosion of Various Metals in Berlin-Dahlem*

(All values in millimol per square meter of surface)

MATERIAL	Experiment November 1944		Experiment February 1945	
	Metal in Corrosion Product	Sulfur-Adhering + Rinsed off	Metal in Corrosion Product	Sulfur-Adhering + Rinsed off
Aluminium	11	8	21	6
Lead	26	12	46	48
Zinc	31	19	53	25
Brass Ms58	32	16	57	24
Copper	32	8	45	11
Nickel	34	23	48	36
Magnesium	76	38	93	lost
Marble	24	19	23	25

* The experiments were carried out on the roof of the State Material-Testing Building in Berlin-Dahlem. Size of tested samples: Approx. 100 x 50 x 1 mm for metals and 90 x 40 x 10 mm for marble.

will depend on the degree of pollution of the former and the salinity of the latter.

B. Individual Metals

Nickel

Reactions involved in the atmospheric corrosion of nickel have been studied extensively by Vernon[16] who has shown that the characteristic fogging of a nickel surface produced by exposure to certain polluted atmospheres is due to the simultaneous presence of sulfur dioxide and water vapor. It also was established that a critical relative humidity of about 70 percent exists below which the metal surface will remain bright indefinitely. Above this critical humidity the surface develops a dull, creamy film which, in its initial stages of formations, consists of a mixture of nickel sulfate and sulfuric acid. With the passage of time, basic nickel sulfate is precipitated as a surface film.

In one case, the formula after 88 days' exposure to an industrial atmosphere was $NiSO_4.0.33\ Ni(OH)_2$. In the preliminary stages of attack the surface film can be removed by wiping. The underlying metal appears unchanged but, as the attack proceeds and the basic sulfate is precipitated, the film becomes adherent and the metal surface develops an etched appearance.

This behavior has been shown to be due to the catalytic oxidation by the nickel surface of atmospheric sulfur dioxide to sulfur trioxide, which absorbs atmospheric moisture to form a dilute solution of sulfuric acid. Reaction with the metal produces a nickel sulfate solution from which precipitation of the basic sulfate can occur after reaction has proceeded for a sufficient length of time. It is well known that sulfur trioxide has a much greater affinity for water vapor than sulfur dioxide and thus its formation will lead more rapidly to an acid liquid film on the surface.

Hudson[3] has shown that in the case of nickel, corrosion will be more severe if the specimens are shielded from the rain than freely exposed to it, and he has attributed this to the hygroscopic nature of the corrosion products which will absorb moisture from the atmosphere to produce a concentrated corrosive solution on the surface. Rain will wash this away, keeping the surface comparatively uncontaminated and free from attack.

These surface changes can be suppressed by pre-exposure of the sample to an atmosphere polluted with hydrogen sulfide. Since pre-heating at elevated temperatures does not prevent fogging as it does the tarnishing of copper, this effect must be due to a poisoning of the catalytic activity of the surface and not to the protective effect of a surface oxide film.

Copper

The atmospheric attack of copper has been discussed in outline in the introduction. Briefly, it was there pointed out that in the presence of hydrogen sulfide the tarnish film contains some sulfide but consists predominantly of oxide. Atmospheric water vapor inhibits the formation of this film. On the other hand, in atmospheres containing sulfur dioxide a critical humidity effect exists. In the absence of water vapor no significant attack takes place, but when the relative humidity exceeds about 63 percent, rapid attack ensues. This results, as with nickel, from the catalytic oxidation of the sulfur dioxide to the trioxide which forms sulfuric acid and attacks the surface. If the concentration of sulfur dioxide is below one percent, as it certainly is in all cases of outdoor exposure, the ultimate corrosion product formed is the basic sulfate.

Vernon and Whitby[17,18] and Vernon[19,20] have carrried out an extensive series of investigations on the characteristic corrosion product, the so-called "Green Patina," that forms on copper when it is exposed out of doors. They examined chemically the patina formed on a series of copper sheathed structures and found that in rural and urban atmospheres it consisted of basic copper sulfate with a small admixture of the basic carbonate. In marine atmospheres some of the basic sulfate is replaced by the basic chloride, which, at distances sufficiently remote from urban areas, may predominate. The basicity of the deposits, depending on their age, may reach a maximum of 3. The corrosion products are:

$Cu\ SO_4\ .\ 3\ Cu(OH)_2$ (Brochantite)
$Cu\ CO_3\ .\ 3\ Cu(OH)_2$ (Malachite)
$Cu\ Cl_2\ .\ 3\ Cu(OH)_2$ (Atacamite)

Vernon has pointed out that this maximum basicity corresponds to coordination formulae and that the well known stability of these compounds accounts for the protective nature of the corrosion product films and the longevity of copper structures.

The development of the patina can be influenced both by prevailing winds and by rain. If the copper bearing structure is remote from the source of sulfurous pollutants the former can carry an adequate supply to the metal surface. Thus, in the example of a church steeple discussed by Vernon, only that side facing the prevailing winds developed a patina. Rain will have a similar effect in that it will bring corrosive gases in the form of dilute solutions to the metal surface.

Zinc

The mechanism of the corrosion of zinc exposed to the open atmosphere has been discussed by Anderson.[21] At low humidities and in the absence of moisture, a visible film grows over the surface of the metal, spreading out from spots where dust particles have fallen on the surface. Below 70 percent, the relative humidity has little influence on these changes but above this value, attack is stimulated and increases with increasing relative humidity.

Anderson[21] has listed the following four characteristics of zinc which will control the reaction on the metal side:

(1) Zinc is attacked by acids at rates which decrease as the pH increases.
(2) Zinc is capable of forming insoluble basic salts when appropriate pH levels are reached.
(3) Zinc has a high hydrogen overvoltage.
(4) Zinc is electronegative to its metal impurities, to hydrogen and to most surface contaminants.

If the film of moisture in contact with the metal surface is comparatively pure then zinc will dissolve as the hydroxide with the evolution of hydrogen. The hydroxide then will react with any atmospheric carbon dioxide dissolved in the water to form the insoluble basic zinc carbonate which will form a protective film. Because of the high hydrogen overvoltage of the zinc, and the neutral nature of the water under these circumstances, the reaction will tend to be slow. However, because aeration in the film of liquid will be excellent, atmospheric oxygen will act as a very efficient depolarizer under these conditions.

In industrial atmospheres the film of moisture can become quite acid through solution of acid effluent gases. Under these conditions the pH can fall as low as 3 and rapid attack which dissolves the zinc and consumes the acid can take place. This results in an increase of pH and a reduction in the rate of attack. At a sufficiently elevated pH a basic zinc salt will precipitate from the solution. This usually is the carbonate, although it can be the sulfate. The precipitated basic salts will form a protective film on the surface of the metal which must be dissolved away before further action can take place.

In his work on the effect of weather on the initial corrosion rate of zinc, Ellis[22] has shown that the initial rate varies markedly between samples exposed at different times and that this variation in initial rate, which depends on the weather conditions at the time of exposure, continues for longer periods of exposure. This is an excellent example of Vernon's general principle that the rate of attack is governed by atmospheric conditions prevailing at the time of first exposure.

Aluminum

The oxide film normally present on aluminum provides excellent protection against atmospheric attack and, in fact, slowly thickens[1,23] when in contact with normal humid atmospheres, thus providing further protection. Laboratory data[23] indicate a mild influence of relative humidity up to 80-85 percent above which the rate of film growth increases appreciably. The presence of gases such as hydrogen sulfide and carbon dioxide in the atmosphere do not influence the protectiveness of this film, but the presence of acid gases such as sulfur dioxide or sulfur trioxide can bring about breakdown of the film with resultant attack of the metal.

Some preliminary unpublished work by the authors on the reaction of aluminum with sulfur dioxide in humid atmospheres gives an insight into the nature of the processes taking place. In this work the weight increment-time curves for superpurity and alloy 3003 aluminum were established for atmospheres of 52, 72 and 85 percent relative humidity containing 0.028 percent by volume of sulfur diox-

ide. The results are presented in Figures 2 and 3 where the weight increment is plotted against time.

At a relative humidity of 52 percent both alloys show weight-increment time curves which approach a limiting value and in which the observed weight increments are of the same order as those observed in the direct oxidation of aluminum in the absence of sulfur dioxide.[23]

At a relative humidity of 72 percent, an induction period appears and while a limiting weight increment is apparent, it is an order of magnitude greater than that observed at the lower humidity; alloy 3003 shows a significantly greater weight increment than the superpurity aluminum at this humidity. The same general observations apply at 85 percent as at 72 percent relative humidity except that a limiting value of the weight increment was not observed within the times studied.

At the lower humidity the metal simply oxidizes, at a rate, and to an extent, determined by the humidity and the characteristics of the metal, with the sulfur dioxide playing no essential role. There is no apparent visual change in the metal surface. At both of the higher humidities the sulfur dioxide plays an essential role as is evidenced by the formation of a white powdery corrosion product which has been identified as $Al_2(SO_4)_3 \cdot 18H_2O$. It is believed that the sulfur dioxide is oxidized to sulfur trioxide on the sample surface which then forms sulfuric acid with the atmospheric moisture and this leads to breakdown of the surface oxide. This process will account for the existence of the observed induction period which would correspond to the time required for film breakdown. As time proceeds a new film consisting of a mixture of the hydrated oxide and sulfate of aluminum will build up and ultimately it will stifle the reaction. The corresponding limiting weight increment was not observed at 85 percent relative humidity, but it probably would be if the observations were taken over a sufficiently long time.

TABLE 2—Composition of Corrosion Products of Magnesium Exposed to an Indoor Atmosphere

Material	Percent Present	
	Time of Exposure in Days	
	412	300
$MgCO_3$	46.5	46.6
$MgSO_4$	9.8	9.7
$Mg(OH)_2$	16.3	15.5
H_2O	27.4	28.2

TABLE 3—Composition of Corrosion Product From Magnesium Exposed to an Outdoor Atmosphere for 217 Days

Material	Percent Present
$MgCO_3 \cdot 3H_2O$	61.5
$MgSO_4 \cdot 7H_2O$	26.7
$Mg(OH)_2$	6.4
Carbonaceous matter	2.5
Siliceous Matter	nil
$Fe_2O_3 + Al_2O_3$	2.9

The experiments were carried out in a closed volume and some hydrogen sulfide was detected at the end of each run. This is probably produced by the reduction of the sulfur dioxide according to the following reaction:

$$2\,Al + SO_2 + H_2O = Al_2O_3 + H_2S$$

although further study will be necessary before the reaction is completely understood.

While the SO_2 concentration studied is higher than that observed in practice (values measured in Great Britain range from 0.000001 to 0.00001 percent by volume[24]) these observations have undoubted relevance to the atmospheric corrosion suffered by aluminum and its alloys. In this regard it is of interest to note that $Al_2(SO_4)_3 \cdot 18\,H_2O$ has been observed in the corrosion product on outdoor structures.[23]

Magnesium

Whitby[25] has studied the corrosion mechanism on magnesium exposed to both indoor and outdoor atmospheres. At constant humidity the weight increment-time curve is linear and the critical relative humidity is about 90 percent. When samples are exposed openly, under fluctuating atmospheric conditions, the weight increment-time curve shows sudden breaks of the form illustrated schematically in Figure 4.

It was found that these breaks were caused by the variable rate of absorbtion of water vapor and carbon dioxide from the atmosphere by the primary corrosion product, magnesium hydroxide. The quantity of each which was absorbed was shown to depend on their concentrations in the atmosphere.

The corrosion product formed during both the indoor and outdoor exposure of magnesium was analyzed with the results shown in Tables 2 and 3.

The presence of magnesium hydroxide in the indoor corrosion products leads to the belief that the hydroxide is the primary corrosion product since it would not form by hydrolysis in the absence of rain water. It is believed that the magnesium hydroxide absorbs carbon dioxide to form the carbonate and this reacts slowly with sulfur dioxide to form the sulfate.

Summary

The atmospheric corrosion of non-ferrous metals depends primarily on the presence of moisture and certain atmospheric pollutants. Atmospheric water vapor, rain, dew or mists will result in the formation of adsorbed or liquid films of moisture on the surface of the metal which can absorb pollutants from the atmosphere with the production of a corrosive solution. In the absence of liquid water a critical relative humidity must in general be exceeded before attack proceeds. In industrial environments the most important pollutant is sulfur dioxide, which is oxidized catalytically to the trioxide and which can then form sulfuric acid with subsequent attack on the metal surface. In the case of certain metals such as magnesium and zinc, carbon dioxide also can play a role. In marine atmosphere air-borne salt or saline spray from the sea can provide the corrosive medium.

While the atmospheric corrosion mechanism of some metals has been worked out in detail, much still remains to be done on individual metals. Analysis of corrosion products can provide very useful information and should be carried out wherever possible, although the leaching effect of rain can complicate interpretation of the results. Such analyses, supplemented by studies carried out in synthetic atmospheres in the laboratory, will

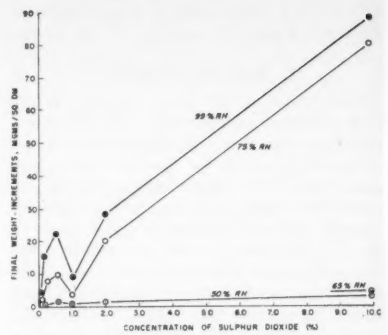

Figure 1—Relation between corrosion and concentration of SO_2 in atmospheres of high relative humidity.

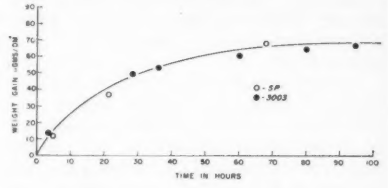

Figure 2—Atmospheric corrosion of super purity and 3003 aluminum at a relative humidity of 52 percent, SO_2 concentration 0.028 percent.

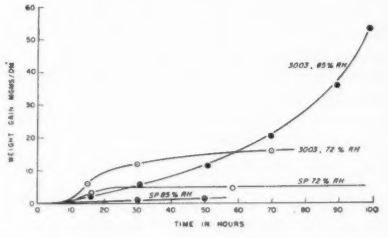

Figure 3—Atmospheric corrosion of super purity and 3003 aluminum at relative humidities of 72 and 85 percent, SO_2 concentration 0.028 percent.

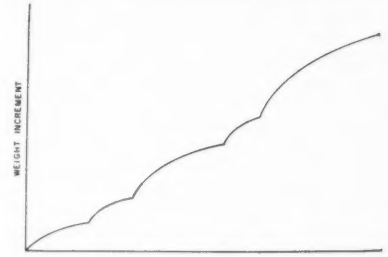

Figure 4—Schematic weight increment time curve for magnesium exposed to an indoor atmosphere.

provide very useful and valuable information.

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Principles and Procedures Employed in the

Design and Interpretation of Atmospheric Corrosion Tests*

Abstract

Principles under which atmospheric corrosion tests are made are much the same as those applicable to other types of testing. Considered, among other things are, uses of accelerated tests, specimen size, location of test sites, differences in atmospheres, differences in metals and influence of alloying constituents.

Mounting procedures, shape of specimens, drainage, influence of rainfall, influence of climatic variations, uses of shelters, reproducibility, test duration, evaluation of results. 2.2.2

By H. R. COPSON*

ATMOSPHERIC corrosion is a truly major corrosion problem. The individual is continually confronted with it in his daily life. Insect screen, hardware, screws, hinges and other metal objects about the home are forever corroding and needing replacement. Bridges, railroad cars, gas tanks and other industrial equipment exposed outdoors require painting and protection. Metallic coatings of zinc, nickel, chromium and other kinds are applied to metal parts to decrease atmospheric corrosion.

The cost of this corrosion and protection is enormous. It has been estimated that the cost of applying paints, varnishes, and lacquers to metals for protection runs around two billion dollars a year.[1] It is evident that there are large potential savings in the field of atmospheric corrosion. Because of this considerable effort has been put into studying atmospheric corrosion and in running atmospheric corrosion tests. There is a large volume of published literature describing atmospheric corrosion tests in many countries and locations throughout the world. In spite of this, it is often desirable to conduct additional tests. It is the purpose of this paper to review what has been learned about atmospheric corrosion testing in the hope that this will prove useful in the design and interpretation of new atmospheric corrosion test programs.

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Corrosion Testing Principles

There is little difference between the principles involved in atmospheric corrosion testing and those involved in other forms of corrosion testing. Corrosion testing may be carried out for several reasons. If the object is to develop theory and mechanism or to develop systematic quantitative information, then laboratory tests where the individual factors can be controlled and studied are in order. If the object is to control the quality of some material or product then some standardized or accelerated test is attractive. More often the corrosion testing is aimed at the evaluation of materials for some specific atmospheric environment, and in this case neither laboratory tests nor accelerated tests are useful.

The most reliable test that can be made is an actual service test. However, service tests are high in cost and take a long time. Usually only a few materials can be tested and there may be interfering influences.

This leaves field testing as the most widespread and probably the best means of obtaining information. In field testing rather small, convenient sized test specimens are simply placed outdoors. However, to obtain really useful results it is necessary to pay attention to many details, such as where the tests are made, how the specimens are designed and mounted, how long the tests are continued and how the results are evaluated. The most important of these details are considered below.

Location of the Tests

In planning atmospheric corrosion tests the first problem is where to conduct the tests. There are many types of atmospheres. They may be classed as industrial, marine, rural, urban, suburban, tropical, etc. As might be expected the corrosiveness of these different types of atmospheres varies greatly.

Figure 1 shows the corrosion behavior of nickel in several atmospheres.[2] Weight losses are plotted against time in years. Altoona, Pennsylvania and New York, New York, are industrial atmospheres. Sandy Hook, New Jersey, is a marine location with some industrial pollution.

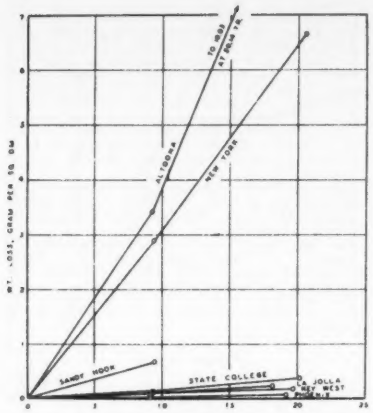


Figure 1—Wt. loss time curves for nickel, material T, in ASTM tests.

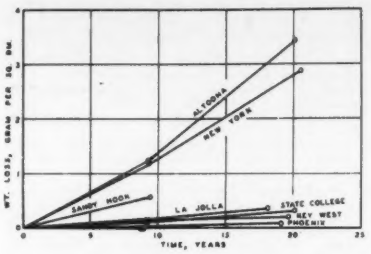


Figure 2—Wt. loss time curves for Monel, material S in ASTM tests.

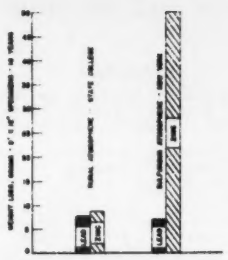


Figure 3—Effect of sulfurous compounds in accelerating corrosion of zinc and protecting lead in the atmosphere.

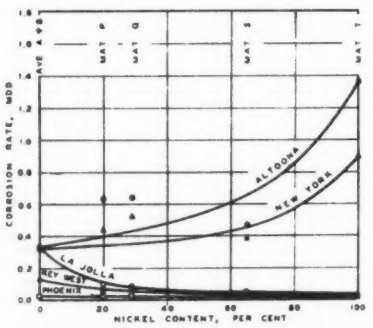


Figure 4—Corrosion rates of nickel-copper alloys in ASTM tests at 20 years.

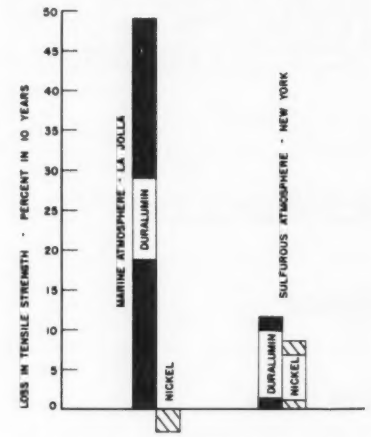


Figure 5—Differences in response of nickel and duralumin to corrosive effects of different atmospheres.

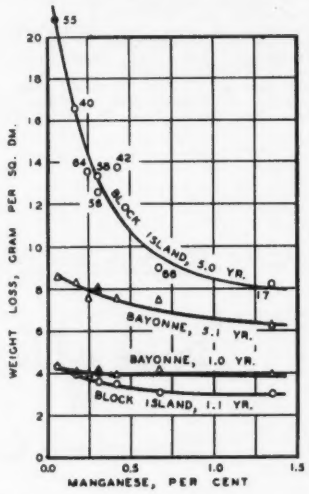


Figure 6—Effect of manganese in low phosphorous 0.3 percent copper steels.

LaJolla, California and Key West, Florida, are marine locations. State College, Pennsylvania and Phoenix, Arizona, are rural locations; Phoenix is quite dry. It is clear that nickel is very resistant to corrosion at marine and rural locations. In severe industrial locations the corrosion was more pronounced.

Figure 2 shows similar data for Monel.[2] The results are quite similar; the industrial locations are much more corrosive than the marine and rural locations. It would be incorrect, however, to assume that this relationship applies for all metals. Even in the case of similar metals like nickel and Monel the relative corrosivity of the different atmospheres varied. For example in the case of nickel, New York was 106 times as corrosive as Phoenix, whereas in the case of Monel the ratio was 43.

Table 1 shows the relative corrosivity of several atmospheres at different locations around the world towards an open hearth iron.[3] Identical specimens were exposed for one year periods. The relative corrosivity varied from 1 to 475—a large spread. Additional tests showed that the relative corrosivity varied considerably from year to year. As with nickel and Monel, dry, rural and unpolluted atmospheres gave the lowest corrosion rates. However, in the case of open hearth iron both marine and industrial atmospheres were corrosive. The difference in relative performance of these three materials in marine locations establishes the impossibility of assigning an arbitrary number to indicate the corrosivity of any location.

The difficulty in classifying any atmosphere is further illustrated by the data at Kure Beach, North Carolina. At a position 800 feet from the ocean the relative corrosivity in Table 1 was rated at 38, whereas at a position 80 feet from the ocean the relative corrosivity was 475, a ratio of over 12 to 1. Many factors such as shelter and time of exposure have similar effects on the ratings.

Further evidence that different atmospheres vary greatly in their corrosivity towards different metals is provided by Figure 3.[3] At the rural location at State College, Pennsylvania neither lead nor zinc corroded very much. However, at the industrial location at New York, New York the corrosion rate of zinc was greatly accelerated. Sulfurous compounds in the air were somewhat protective towards lead but increased the corrosion of zinc.

Additional evidence of this sort is provided by Figure 4. This shows the corrosion rate of some nickel-copper alloys in some ASTM tests.[2] The important point to notice is that at the marine locations of LaJolla and Key West, nickel had a

TABLE 1—Relative Corrosivity of Atmospheres At Different Locations[3]
(As determined using open hearth iron specimens, 2 x 4 x ⅛ inch)

Location	Type of Atmosphere	Avg. Wt. loss, grams in 1 Year	Relative Corrosivity
Khartoum, Egypt	Dry Inland	0.16	1
Abisco, North Sweden	Unpolluted	0.46	3
Singapore, Malaya	Tropical Marine	1.36	9
Daytona Beach, Fla.	Rural	1.62	11
State College, Pa.	Rural	3.75	25
South Bend, Pa.	Semi-Rural	4.27	29
Miraflores, Canal Zone (Pacific Side)	Tropical Marine	4.5	31
Kure Beach, N.C. (800 ft. from ocean)	Marine	5.78	38
Sandy Hook, N.J.	Marine, Semi-Ind.	7.34	50
Kearny, N.J.	Industrial, Marine	7.75	52
Vandergrift, Pa.	Industrial	8.34	56
Pittsburgh, Pa.	Industrial	9.65	65
Frodingham, British Isles	Industrial	14.81	100
Daytona Beach, Fla.	Marine	20.43	138
Kure Beach, N.C. (80 ft from ocean)	Marine	70.49	475

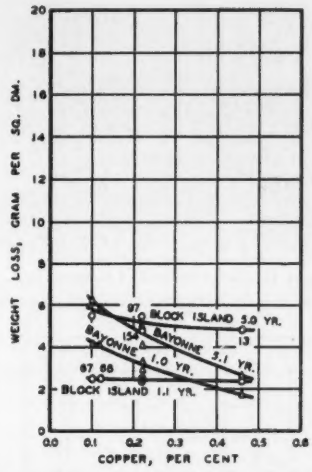


Figure 7—Effect of copper in steels of Group V with high phosphorous, chromium, and silicon.

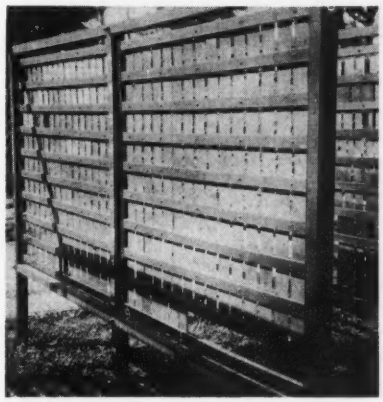


Figure 10—Vertical wooden racks at Bayonne, N. J.

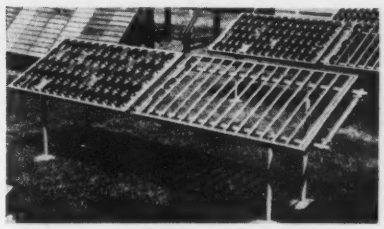


Figure 8—Inclined metal racks at Bayonne, N. J.

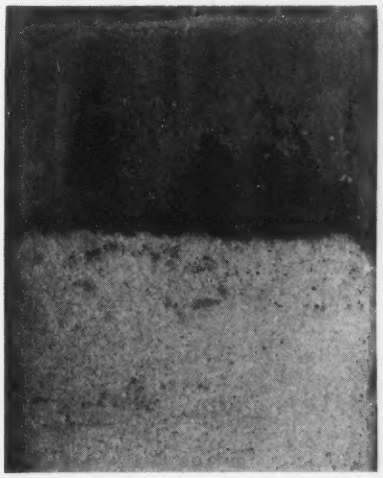


Figure 11—Accelerated attack on brass associated with drainage from a solder coating. Specimen exposed on inclined racks at Bayonne for 4.3 years.

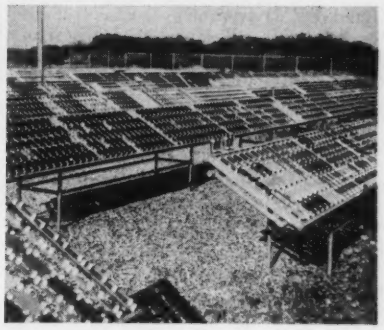


Figure 9—Atmospheric test racks at Kure Beach, N. C.

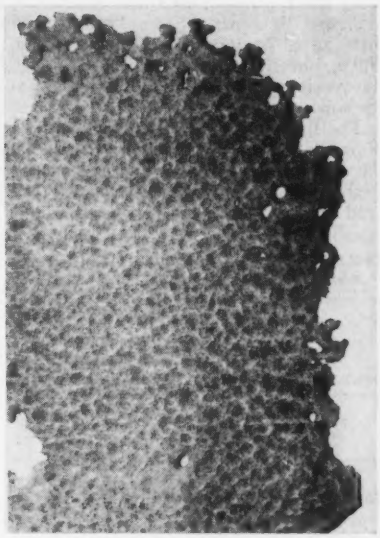


Figure 12—Corrosion at edges of steel specimen associated with washing action of rain. Specimen exposed on inclined racks at Block Island for 3 years.

lower corrosion rate than copper, whereas at the industrial locations of Altoona and New York, nickel corroded faster than copper.

This type of behavior is important in galvanic couples. In accord with these results in some ASTM tests,[4] nickel seemed cathodic to copper in marine locations and anodic to copper in industrial locations. Some tests at Bayonne, New Jersey have confirmed that copper is more noble than nickel at industrial locations.[5] It is thus evident that the polarity of galvanic couples may change with location.

A further comparison between different metals is provided by Figure 5.[3] This shows the loss in tensile strength of specimens of nickel and of an aluminum alloy in a marine and an industrial atmosphere. While both atmospheres were destructive to this particular aluminum alloy, only the industrial atmosphere had an appreciable effect on nickel. It should be remarked that the aluminum alloy chosen to illustrate the point under discussion is not representative of most corrosion resistant aluminum compositions and is not ordinarily used in corrosive atmospheres without supplementary protection.

This brings up the observation that in addition to considering the different metals it is necessary to consider their alloying constituents. This can be illustrated by the effects of some of the alloying elements in steel. Figure 6 shows the effect of manganese content on the corrosion rate of steel at two locations.[6] Block Island, Rhode Island, is a marine location and Bayonne, New Jersey is an industrial location. At Block Island, manganese is beneficial in low-phosphorus, 0.3 percent copper steels, and the amount of benefit seems to be increasing with time. At Bayonne, manganese had no detectable effect on the weight losses of these steels at one year, but by five years some benefit was showing.

A little copper in steel is generally very beneficial in decreasing atmospheric corrosion. However, the effect of copper varies with location and with the composition of the steel. This is illustrated by Figure 7[6] which shows the effect of copper in some complex low alloy steels. For these particular steels copper contents above about 0.1 percent were without much effect on the weight losses at Block Island. On the contrary, at Bayonne weight losses decreased steadily with copper content up to 0.5 percent at least. It is evident that copper additions were more beneficial in the industrial Bayonne atmosphere then in the marine Block Island location.

Owing to the fact that the beneficial effects of alloying elements in steel vary with location, it is not surprising that different steels might be rated in reverse order at two locations. This is illustrated by Table 2,[6] which gives the weight losses of three steels at Bayonne and Block Island. The straight nickel steel would be rated best at the marine Block Island location, whereas in the industrial Bayonne atmosphere additional alloying with chromium and copper gave improved results.

The conclusion from these observations is that it is desirable to conduct atmospheric corrosion tests in as many different atmospheres as possible. Otherwise the data obtained are incomplete and may even be misleading. As a minimum it would seem that tests should be conducted in marine, industrial and rural, or suburban atmospheres. If the interest is limited to some particular situation, then the tests should be conducted in the atmosphere or location of interest and not somewhere else. Results obtained at one location will not necessarily apply at some other location.

Mounting The Specimens

In planning atmospheric corrosion tests, after the test sites have been se-

lected, the next problem is how to mount the specimens. Of course there are many possible ways of doing this. In this country there has been a tendency to standardize on the type of rack being used by the American Society for Testing Materials.

Figure 8 illustrates some of these racks as installed at Bayonne, New Jersey. Here the legs and supports are galvanized iron pipe. The frame holding the specimens is Monel. The specimens are mounted between porcelain insulators attached to the frame. In the figure the specimens are 4 by 6 inches in size, although the racks can accommodate quite a range of dimensions. The specimens are exposed at an angle of 30 degrees from the horizontal and face south.

Figure 9 shows a large number of these racks as installed at Kure Beach, North Carolina. The groundward side of the specimens is sheltered somewhat from the sun and rain.

The British often prefer a vertical type of rack, but even here the two sides of the specimens do not receive the same exposure. Figure 10 shows a vertical rack used at Bayonne, New Jersey. Here the specimens are mounted in wooden slots. It is necessary to keep the specimens insulated from each other.

There will be some difference in corrosion behavior on the two types of racks. In addition to the angle of exposure, the direction of exposure and the elevation above ground level will have some effect. The magnitude of these effects will vary with location and with the source of any atmospheric pollution. Generally these effects are relatively minor, but they may change the rate of attack by as much as 25 to 40 percent.[6] It is not safe to make direct comparisons between results obtained on different types of racks.

While bold exposure of sheet specimens may suffice to give a general idea of the atmospheric corrosion resistance of the material, nevertheless many important and damaging effects will be missed. Nothing will be learned about the effect of specimen size and shape, about the effects of shelter, crevices and drainage areas, or about galvanic effects. The effect of these variables on corrosion can be quite large.

The shape influences the amount of air-borne pollution which impinges on the specimen.[7] It is axiomatic that the corrosion rate of metals in the atmosphere varies with the amount of pollution present. Therefore the corrosion rate can be expected to increase with the amount of contamination actually coming in contact with the specimen. This is controlled not only by the degree of pollution present but also by the shape and form of the specimen.

A good illustration of this is the difference in corrosion rates of wire and sheet specimens. In Table 3 wire corroded 2 to 4 times faster than sheet.[8] It seems to be a general rule that corrosion rate increases as the wire is made smaller in diameter. This is due largely to the effect of better air circulation around the wire which causes more atmospheric pollution to come in contact with it than is the case with sheet specimens. What counts is the amount of pollution received by the specimen per unit of area. A corrugated specimen would be expected to show a lower rate of penetration than a sheet specimen because it would receive less pollution per unit area. Evidently shape can have a big effect on the corrosion.

Another factor which can affect the amount of pollution per unit area is associated with drainage. For example if rain lands on some inert material such as shingles and then drains onto metal, the amount of corrosion occurring on the metal beneath the edge of the shingles can be particularly severe. The corrosive action of all the pollution accumulated by a large area of shingles is then concentrated on a small area of metal.[9]

This effect is illustrated by Figure 11. Here a sizeable area of a brass specimen was coated with solder, and the specimen was then exposed outdoors on an inclined rack for 4.3 years in the industrial atmosphere at Bayonne, New Jersey. Like lead, solder develops a protective film in sulfurous atmospheres. Accordingly the corrosive constituents landing on the solder were not consumed, but drained down the specimen and reacted with the brass at the edge of the solder. This drainage effect extended over an area about one inch wide and the deepest local penetration of the brass amounted to 0.007-inch.

The washing action of rain may sweep the corrosives to the lower edge of the

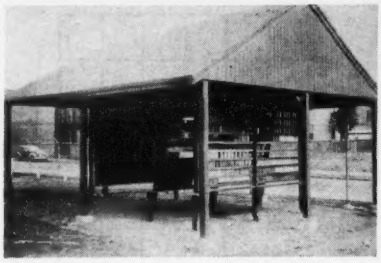


Figure 13—Sheltered racks at Bayonne, N. J.

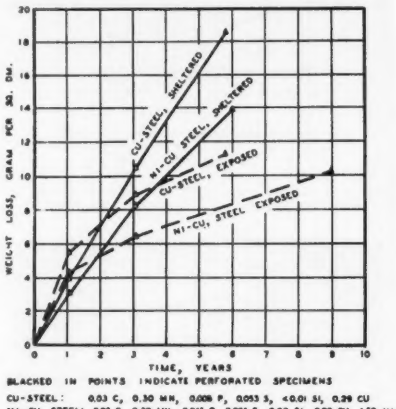


Figure 14—Effect of shelter on specimens exposed vertically at Bayonne.

TABLE 2—Effect of Atmosphere on Relative Merit As Indicated by Corrosion Rates[4]

Steel No.	Analysis, Percent			5 Yr. Wt. Loss, g/sq. dm.	
	Ni	Cr	Cu	Block Island	Bayonne
46	2.2	..	..	5.37	5.30
30	1.2	0.7	..	5.53	5.10
148	1.0	..	0.9	5.89	3.89

TABLE 3—Relative Corrosion Rates of Wire and Sheet[8]

Values of average thickness of corroded layer expressed as inches per year x 10^{-4}. Duration of test, 1 year. Corrosion rates of wires determined by electrical resistance method. Corrosion rates of sheets determined by weight losses. Average of five stations.

Material	Wire (0.048 in.)	Sheet	Ratio
80/20 Nickel-chromium	1.67	0.47	3.6
Arsenical copper	3.05	1.16	2.6
H.C. copper	2.88	1.31	2.2
70/30 Nickel-copper	4.08	1.34	3.0
Cadmium-copper	3.14	1.35	2.3
Nickel	3.78	1.40	2.7
Aluminum bronze	3.23	1.50	2.2
Silicon bronze	3.11	1.57	2.0
80/20 Nickel-copper	2.84	1.62	1.8
Tin bronze	2.47	1.74	1.4
70/30 brass	4.42	1.91	2.3

TABLE 4—Atmospheric Corrosion of Some Stainless Steels[2]
Exposed vertically at Bayonne, N. J., for 11.88 years

Type No.	Composition, Percent			Sheltered		Exposed	
	Ni	Cr	Other	Mdd	Pit Depth Mil	Mdd	Pit Depth Mil
304	8.9	18.4	.....	0.062	7	Nil	Nil
321	9.7	18.7	0.48Ti	0.038	6	Nil	Nil
347	11.2	18.6	0.78Cb	0.045	6	Nil	Nil
316	13.1	17.8	2.8Mo	Nil	<1	Nil	Nil
317	14.1	18.6	3.5Mo	Nil	<1	Nil	Nil
308	10.7	20.4	.....	0.017	7	Nil	Nil
309	13.6	23.6	.....	0.001	1	Nil	Nil
310	19.8	24.1	.....	0.008	6	Nil	Nil
430	0.3	17.1	.....	0.030	8	Nil	Nil

TABLE 5—Effect of Time of Exposure in Bringing Out Differences Between Low Alloy Steels[6]

Steel No.	Wt. Losses at Bayonne, g./sq. dm.		
	1.0 Yr.	5.1 Yr.	Increase
68	4.09	5.91	1.82
53	3.96	6.05	2.09
67	4.10	6.20	2.10
7	4.03	6.88	2.85
66	4.13	7.46	3.33
118	4.02	7.72	3.70
43	4.07	7.98	3.91
41	4.07	8.03	3.96
58	4.00	8.05	4.05
40	4.08	8.30	4.22

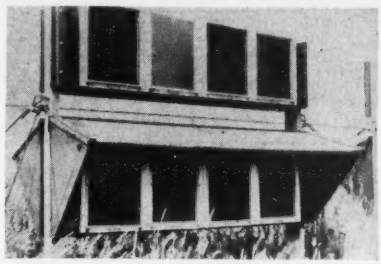


Figure 15—Typical exposure rack for insect screen.

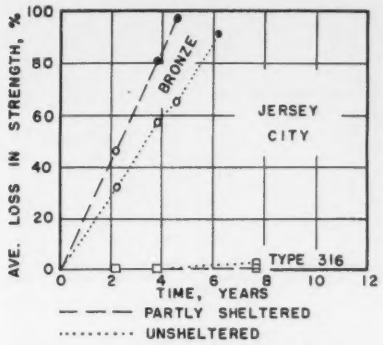


Figure 16—Atmospheric corrosion of bronze and Type 316 stainless steel insect screen.

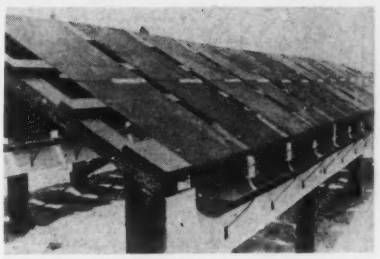
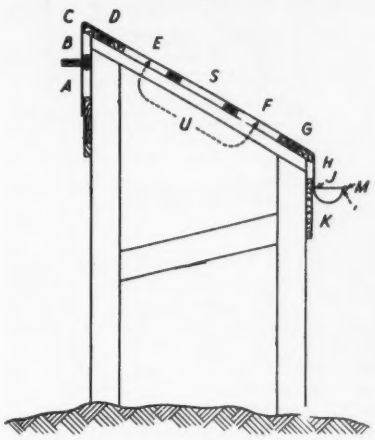


Figure 17—Simulated roofing racks



(*A*) Vertical surface, partly sheltered from direct impingement of rain.
(*B*) Vertical surface, boldly exposed to rain.
(*C*) Sharp bend in sheet.
(*D*) Area having lower surface in contact with wood.
(*E*) and (*F*) Plane inclined surfaces.
(*G*) Area having lower surface in contact with wood.
(*H*) Vertical surface, boldly exposed.
(*J*) Area of water-line attact when trough is full of rain water.
(*K*) Area completely submerged when trough is full of rain water.
(*L*) Area of water-line attack.
(*M*) Trough bead.
(*S*) Lock seam, one-half soldered, one-half not.
(*U*) Fully sheltered lower surfaces.

Figure 18—Construction details of simulated roofing rack.

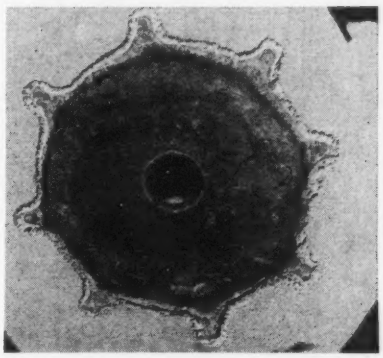


Figure 19—Steel core in magnesium casting after 9 years in a marine atmosphere.

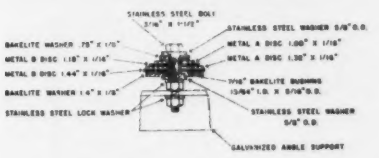


Figure 20—Atmospheric galvanic couple test assembly.

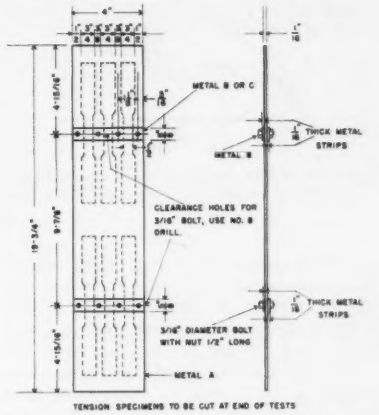

METAL B OR C
THICK METAL STRIPS
METAL B
CLEARANCE HOLES FOR 3/16" BOLT, USE NO. 8 DRILL.
THICK METAL STRIPS
3/16" DIAMETER BOLT WITH NUT 1-1/2" LONG
METAL A
TENSION SPECIMENS TO BE CUT AT END OF TESTS

Figure 21—Plate and fastening type atmospheric galvanic couple test specimen.

specimen and cause accelerated attack there. Figure 12 shows an instance where this occurred on a steel specimen exposed to a marine atmosphere.[10] Not only was the attack greater at the lower edge, but the prevailing direction of the wind during rainstorms concentrated the attack at the left or western edge. The attack was severe also under insulators at the right edge where the washing action of rain was impaired. Generally the washing action of rain is beneficial to steel. It is only harmful where it causes the corrosives to accumulate, as at the edges in the figure. Presumably this edge effect might increase with specimen size.

Sheltered areas receive no washing action by rain, and accordingly corrosion at sheltered areas may be quite different from that at exposed area. It is possible to study the effect of shelter by placing the test racks under a roof. Figure 13 shows how that was done at Bayonne, New Jersey. The essential features are to provide free circulation of air, but to prevent rain from washing the specimens. Shelter affects different metals differently and its effect may vary with location. Usually shelter is harmful to iron and beneficial to zinc.

Figure 14 shows the effect of shelter on two steels exposed vertically at Bayonne, New Jersey.[6] At one year the weight losses were greater on the exposed specimens, probably because they were wet more often. However, by three years conditions had reversed and from then on weight losses were greater on the sheltered specimens. The washing action of rain had helped the exposed specimens build a more protective rust coating. Thus it makes a difference how long the tests are continued. Also it makes a difference how the results are evaluated. The exposed specimens were pitted to about the same depth as the sheltered ones and perforated just as soon.

Table 4 shows the effect of shelter on some stainless steel specimens exposed vertically at Bayonne, New Jersey.[2] Boldly exposed, all these stainless steels were essentially unattacked even after 12 years of exposure. In the sheltered exposure the only stainless steels to remain unattacked were the molybdenum-containing varieties, Types 316 and 317, although the high-chromium Type 309 was fairly good. All the others developed rust spotting which spread inwards from the edges until 50 percent or more of the surface appeared rusty. Pit depths in the table were measured on the plain surfaces. These specimens were exposed on the vertical wooden racks shown earlier. The lower edge of the specimens was inserted in a wooden slot. Pitting was deeper in this crevice than on the plain surfaces. It appears that corrosion may often be more severe in crevices than elsewhere.[2]

Owing to the importance of such factors as shelter, crevices, drainage areas and specimen size and shape, it often is desirable or even necessary in atmospheric corrosion testing to simulate the service conditions as closely as possible. This was done, for example, in testing insect screens.[11] Figure 15 shows a typical test rack. Actual insect screen was used. The screen frames were mounted on the solid wall of the rack in such fashion as to form a box-like space about three inches deep behind each screen. This permitted air to circulate but prevented wind from blowing through. Some of the screens were boldly exposed and some were sheltered to simulate conditions on porch windows or under eaves.

Some results from these tests are shown in Figure 16. The Type 316 stainless steel proved to be practically unaffected when exposed as insect screen at a severe industrial location in Jersey City, New Jersey. In the case of bronze screen, shelter had some effect in decreasing the life. In the case of some other screen

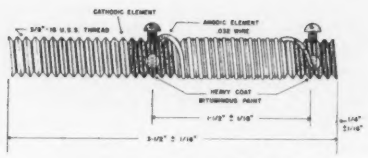


Figure 22—Bolt and wire type atmospheric galvanic couple test specimen.

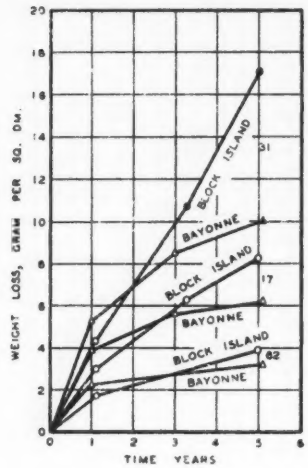


Figure 25—Weight loss versus time curves for three steels. Blacked in points indicate perforated specimens.

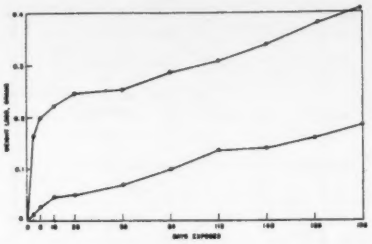


Figure 23—Time-corrosion curves of two samples of sheet zinc showing high and low initial rates of corrosion.

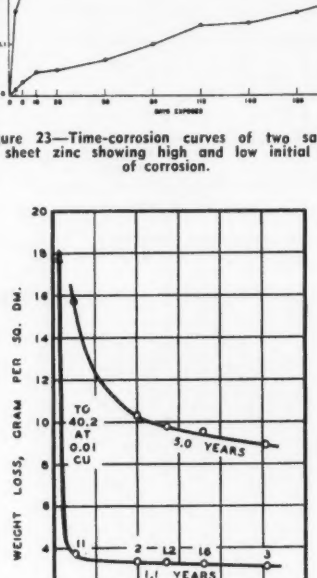


Figure 26—Weight loss versus copper content of Group III, high phosphorous, copper steels at Block Island. Blacked in point indicates perforated specimen.

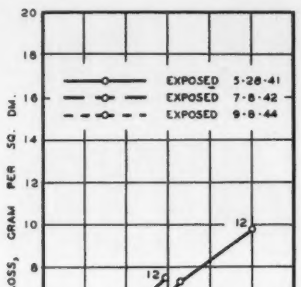


Figure 24—Change in corrosivity of Block Island atmosphere with time.

materials, such as Type 302 stainless steel and aluminum, shelter may have an even bigger effect, with the difference in the performance of sheltered and exposed screen being quite large. The short life of sheltered screen would not have been predicted from tests on sheet specimens on bold exposure. The physical structure of screen cloth, with its multiplicity of contacts and the effects of wire intersections in retaining dirt and stabilizing droplets of liquid, make it uniquely susceptible to the detrimental effect of shelter from rain.

Another instance where service conditions were simulated was in the case of some tests on roofing materials.[12] The racks used for these tests are shown in Figure 17. They provided for inclined sloping areas, a lock seam, a soldered seam, sharp bends and for what might be called an alternate immersion exposure provided by an undrained rain trough. They also provided for vertical areas and for sheltered areas, as perhaps can be seen better by the sketch in Figure 18.

A number of low alloy steels were included in the tests. Perforation always occurred first in the rain troughs, next in the completely sheltered areas marked A, and then at B. The inclined surfaces E and F were next to go, with positions D and G holding up longest.

Special tests will be required to study special conditions, such as are involved in galvanic corrosion, stress corrosion cracking, abrasion and protective coatings. Galvanic corrosion in the atmosphere usually does not extend very far from the line of contact. An idea of this is provided by Figure 19.[3] This shows the corrosion of an assembly of magnesium cast around a steel hub which was exposed for nine years at Kure Beach, North Caroline, 80 feet from the ocean. The galvanic acceleration of the corrosion of the magnesium did not extend more than about 3/16 inch from its line of contact with the steel and the galvanic protection of the steel was restricted to a similar difference. This means that area ratios are not important in galvanic corrosion testing in the atmosphere and that test specimens can be designed without worrying about this factor.

TABLE 6—Effect of Time of Exposure on Relative Merit of Low Alloy Steels[5]

Steel No.	Analysis, Percent			Wt. Loss at Bayonne, g./sq. dm.		
	Ni	Cu	Cr	1.0 Yr.	3.0 Yr.	5.1 Yr.
28	1.5	...	...	3.29	4.56	5.82
60	1.0	1.0	...	3.49	4.49	5.32
30	1.2	...	0.7	3.70	4.42	5.10

Figure 20 shows a galvanic corrosion test assembly used by ASTM Committee B-3.[13] In this assembly, each of the two middle specimens has a specimen of the other metal on each side of it. Only the two middle specimens are used in appraising the results. The exposed areas of these specimens are equal.

Figure 21 shows another type of galvanic corrosion test assembly. This is made by attaching a strip of one metal to a panel of another by riveting, bolting, or spot welding. After exposure, tension test specimens can be cut from the assembly. The area of greatest galvanic corrosion is located at the reduced section of the test piece.

Still another type of galvanic corrosion test assembly is shown in Figure 22. This was developed by the Bell Telephone Laboratories. Here the less noble metal is in the form of a wire wound in the grooves of a threaded specimen of the more noble metal and held in good electrical contact with it by set screws covered with a protective coating. Galvanic corrosion damage is favored by the high ratio of contact surface to mass of the wire specimen.

It is clear that many types of specimens can be used for galvanic corrosion testing. Many types of specimens can also be used for studying stress corrosion cracking in the atmosphere. The main feature here is to apply a tensile stress, which may be in the form of a constant load or of a constant deformation. Many suitable specimens were described in a symposium on stress corrosion cracking[14] and these will not be discussed further here. The design of suitable specimens seems to be limited only by the ingenuity of the experimenter.

There are other special conditions which require special tests. For example if abrasion of the metal surface is taking place, the tests must be designed to take this into account.[15] An instance where abrasion would be important is in the operation of coal cars.[12] Special tests also may be required in studying the performance of protective coatings, both metallic and organic. Enough has been

said, however, to show the importance of care in the design and mounting of atmospheric test specimens. The value of the results obtained will be in proportion to the amount of care which has been expended on these details.

Reproducibility

Before the tests are started some consideration should be given to their reproducibility. Because weather is far from constant, the weather following the start of a test might be unusually wet or unusually dry or have some other peculiarity. This can have a substantial effect on the corrosion which is observed.[3]

Figure 23, for example shows some data obtained on zinc.[16] The two weight loss time curves represent different starting dates. Obviously there was a large difference in the initial corrosion rates and the effect of this difference persisted over quite a time.

Figure 24 shows some data on two steels at Block Island, Rhode Island. Here the corrosivity of the atmosphere seems to be increasing with time. Some years can be much more corrosive than others and the differences can be much larger than indicated by the figure.

There is not much that can be done about this situation. Attempts have been made to correlate corrosion rates with atmospheric conditions. The humidity, rainfall, temperature, pollution, wind direction, length of time wet and other factors have been studied, but so far it is not possible to make measurements of atmospheric conditions and predict accurately how these will affect the corrosion of test specimens.

There is one way out of this dilemma and that is to expose some control panels each time a new test is started. These will give some idea of how the atmospheric conditions have changed and will permit comparisons between tests started at different times. Without the controls, such comparisons might lead to erroneous conclusions.

In addition to putting out controls, some thought should be given to statistical considerations. This is beyond the scope of the present paper, but certainly the specimens should be exposed in replicate. How many specimens are exposed will depend on the nature and purpose of the tests.

Duration of the Tests

Once the test sites have been selected and the design and manner of mounting the specimens has been decided, the next problem is how long to continue the tests. Ideally the tests should be continued as long as something more can be learned. Usually this requires a long duration, particularly if the rate of attack is changing with time. This can be illustrated by some data on low alloy steels.

Figure 25 shows some weight loss versus time curves for three low alloy steels at two locations. Initially they all started corroding at a relatively fast rate.[6] However, they soon developed protective rust coatings and the corrosion rate decreased with time. This effect was more pronounced on the more highly alloyed steels, namely steels No. 17 and No. 62. Thus the benefit of alloying increased with time. Also this effect was more pronounced at the industrial Bayonne location than at the marine Block Island location. At one year it might have been concluded that the industrial atmosphere was the more corrosive, but the reverse was true by five years. Early in the tests the differences in weight losses were not great, but by five years they were pronounced.

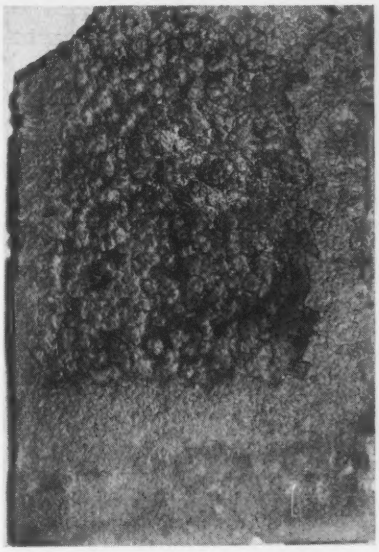


Figure 27—Specimen of steel corroded to destruction without perforation at Kure Beach, N. C.

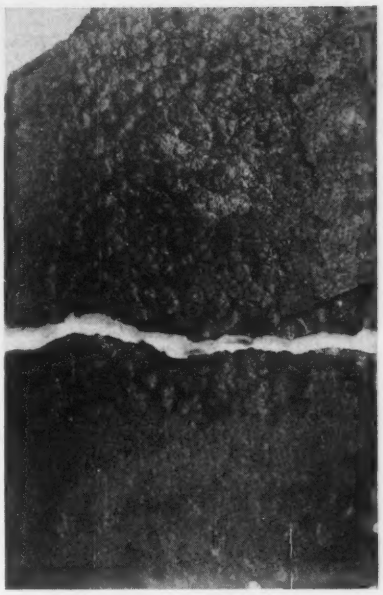


Figure 28—Same specimen as Figure 27 after slight bending.

Even after one year steels which differ greatly in ultimate merit may have practically identical weight losses. This is indicated in Table 5 for 10 steels at Bayonne.[6] During the first year these 10 steels corroded at the same rate, but from the first to the fifth year the best steel of this group corroded less than half as fast as the worst. The better steels in this group were higher in alloy elements, chiefly in manganese, chromium and phosphorus.

Much the same point is brought out by Figure 26. This shows the effect of copper content on some high phosphorus steels at Block Island, both at 1 and 5 years.[6] A little copper was very beneficial, but the effect of additional copper was less marked. After one year it might be concluded that there was little benefit to be derived by more than 0.05 percent copper in these steels. However, from one to five years steel No. 11 with this amount of copper corroded more than twice as fast as steel No. 3 with 0.5 percent copper. At five years there was steady improvement with increasing copper content. The inflection point in the curve was less pronounced and had shifted toward higher copper content.

As might be expected from these changes in relative corrosion rates with time, it is possible for the relative merit to change with time. This is illustrated by Table 6 which shows the weight loss data on three low alloy steels at Bayonne, New Jersey.[6] After one year the best steel seemed to be the straight nickel steel, No. 28. By three years both the nickel-copper steel, No. 60 and the nickel-chromium steel, No. 30 had surpassed the straight nickel steel, and by five years the trend was even more pronounced.

These results indicate that there is no definite answer to the question of how long the tests should be continued. This will depend partly on the objectives of the tests. It is clear, however, that it is desirable to remove specimens after several time intervals, and to obtain corrosion rate versus time curves. It is also clear that the tests probably will run for several years. It is not uncommon for atmospheric corrosion tests to run for 10 or 20 years or even longer.

Accelerated Tests

Owing to the prolonged duration of atmospheric exposure tests there have been a number of attempts to develop accelerated tests.[3] The idea behind these tests is to produce the same results in a short time as normally require years of outdoor exposure. The Acid Test was an early example of this and the Salt Spray Test a more recent example and there have been others. Such tests may be useful in special situations, but it should be obvious that reproducing the atmospheric corrosion behavior of metals in any one accelerated test is an impossibility. There is too much variation among atmospheres, between near-by locations in the same atmosphere, between different metals and alloys and alloying components, between different shapes and forms of test specimens and the manner of exposure and between different exposure periods. In other words there are so many possible answers depending upon circumstances that an accelerated test which provides one answer or even a

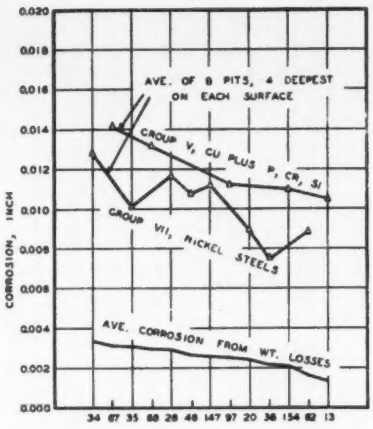


Figure 29—Group V and VII steels arranged in order of weight losses at Bayonne at 5.1 years.

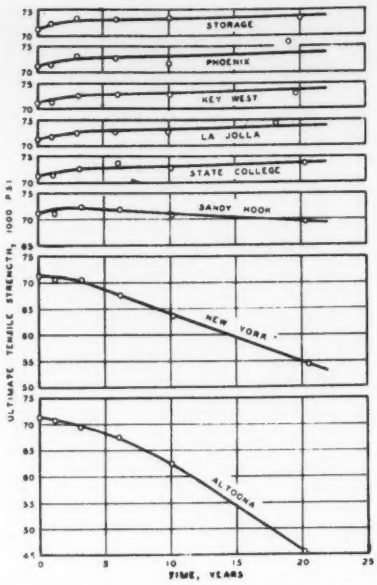


Figure 30—Strength vs time for nickel tensile specimens.

series of answers cannot be expected to tell the whole story. It is a safe prediction that outdoor atmospheric exposure tests will continue to be conducted for some time to come.

Evaluation of the Results

One further problem which should be settled before atmospheric tests are started is how the results should be evaluated. Many methods have been used, such as appearance ratings, weight losses, pit depths and losses in strength. These different methods do not necessarily lead to the same conclusions. Certainly some method should be used which gives the information wanted. Preferably several methods should be used so that a complete picture may be obtained.

When, as in the case of decorative coatings, appearance is the only important consideration, appearance ratings have been defined.[17] For determining durability, however, appearance can be deceptive. There may be corrosion underneath protective coatings and there may be corrosion beneath the surface of apparently solid metals. Also corrosion products can obscure or hide severe damage. For example in some early tests on steel, the time to disintegration or perforation was used as the criterion of failure. The rust coating hid early perforations and to a considerable extent the life of the rust was included in the life of the steel.

The seriousness of this is illustrated by Figures 27 and 28.[3] Figure 27 shows a rusted specimen of steel as it was taken from a test rack. There is no visible evidence of perforation. Figure 28 shows the same specimen after slight bending. It broke easily and further bending indicated that there was really nothing left but stratified layers of compact rust. The metal itself was completely corroded away.

Weight gains have been used to get some idea of the amount of corrosion taking place without destroying the test specimens. However, these are not very satisfactory because there is no way of knowing how much of the corrosion products have been lost. Weight gains should be supplemented with analysis of the corrosion products.

Another non-destructive method is to measure changes in electrical resistance. This requires the use of wire or strip specimens. It has the advantage that measurements can be made at frequent intervals so that the progress of corrosion can be followed closely. It has the disadvantage that it may not indicate pitting or other serious forms of damage.

Where possible it is preferable, as already indicated, to expose specimens in replicate and to remove some of these at selected intervals. Destructive measurements then can be made on the removed specimens and they are never returned to the test racks. The measurements might be of weight losses, pit depths, or losses in strength and it often is helpful to make all of these measurements.

Where corrosion is uniform, weight losses are a highly satisfactory means of evaluating the results. Most of the corrosion data which have been presented were obtained as weight losses. To obtain weight losses the corrosion products must be removed and this sometimes is difficult. Care must be taken to have negligible attack in cleaning. Satisfactory methods are described in the literature and these should be referred to. Where metals of different density are involved, it is instructive to convert the weight losses into penetration in inches or mils per year. Weight losses will tend to be favorable to a light metal like aluminum and inches penetration to a heavy metal like lead.

Once a specimen has been cleaned of its corrosion products it should be examined carefully for local attack or pitting. Large pits will be apparent to the eye, but in addition the specimen should be examined under a low power microscope, because the pits may be small in diameter even though appreciably deep. Where corrosion is not uniform the depth of attack should be measured. A pointed probe attached to a dial gage may suffice to do this. Another method is to calibrate a microscope and focus alternately on the pit bottom and on the surrounding surface. Neither of these methods may reach the maximum depth of attack if the pit undercuts the surface, or if it follows an intergranular path. In this case microexamination of polished cross sections may be necessary to reveal the full depth of attack.

Weight losses and depths of local attack do not necessarily parallel one another. Figure 29 provides an illustration of this in some tests on low alloy steels.[6] Here the steels of two groups, called V and VII, are arranged in the order of their 5-year weight losses at Bayonne. Both the average corrosion from weight losses and the pit depths are plotted. The nickel steels of group VII consistently have lower pit depths than the steels of group V. Steel No. 13 which would be rated best on the basis of weight losses is surpassed by several steels on the basis of pit depths. Probably this behavior is connected with the number of points of attack, with steels containing chromium tending to have fewer but deeper pits and steels containing nickel tending to have more but shallow pits. Measurements with flat micrometers showed that there had been no loss in maximum thickness of these steels. All the corrosion occurred at the pits. For a thin steel where perforation may be relatively fast, pit depths are more important than weight losses.

For these same steels subsequent measurements[16] showed that there was practically no increase in pit depths from five to nine years even though weight losses showed that corrosion was continuing. Presumably new pits were forming and old ones were widening. The increased weight loss was due to these factors rather than to deepening of existing pits. The corrosion was spreading laterally over the surface.

In cases such as this the rate of loss in strength of the material might be rapid for the first year or so and then drop to a very low value. Furthermore the magnitude of the loss in strength could be minimized by making the material thicker. For these reasons it is useful to measure the tensile strength of atmospheric test specimens. In the case of aluminum alloys measurements of the tensile strength and of pit depths are preferred over weight loss measurements.[19]

Figure 30 shows some tensile test data on nickel which were obtained during 20-year atmospheric exposure tests.[2] The specimens were exposed as pre-machined standard tension test specimens and pulled at the times indicated in the figure. In tests of this kind it is necessary to have a storage set of tensile specimens, because there may be some increase in strength with time due to age-hardening effects. This occurred to a small degree in the case of nickel, but much larger effects may be observed with other metals and alloys. Using tensile strength as the criterion of damage it is evident that there was negligible corrosion of nickel through 20 years at the marine and rural

locations of Phoenix, Key West, LaJolla and State College. On the other hand at the severe industrial locations of New York and Altoona, there was appreciable loss in strength.

One objection to exposing pre-machined tension specimens is that corrosion at the edges may produce notch effects which makes the losses in strength and ductility unduly large. One way around this difficulty is to expose strip specimens and machine them into tensile specimens just before pulling. Tensile testing has an advantage in that specimens do not have to be cleaned before pulling. Such tests are very useful in the case of insect screen where the chief concern is with pitting and wire perforations. Tensile testing also will reveal sub-surface attack, such as intergranular corrosion or dezincification.

The information obtained by tensile tests, by pit depths, by weight losses, visual examination and other observations all supplement each other, and lead to a rounded picture of atmospheric corrosion which cannot be obtained from one set of observations alone. Because atmospheric corrosion data takes a long time to acquire, it seems worthwhile to make as many kinds of measurements on the test specimens as possible.

Conclusion

This article has tried to stress the principles and procedures employed in the design and interpretation of atmospheric corrosion tests. It is predicted that outdoor atmospheric exposure tests will continue to be conducted for some time to come. It is hoped that this discussion has shown how to make these tests more useful and informative.

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Deterioration of Materials In Polluted Atmospheres*

Abstract

A brief summary of the chemical and physical aspects of polluted atmospheres. More specificially it deals with the detrimental effects of air pollutants on materials other than living tissue. In addition to corrosion of metallic surfaces, the physical and chemical principles involved in the damage to such materials as paint, glass, stone, rubber, and fibers by man-made air contaminants are discussed. 4.2.8

By JOHN E. YOCOM*

A NUMBER of undesirable effects of air pollution are well recognized by those who have spent considerable time in densely populated areas. Some of the more obvious of these effects are:

1. Annoyance to the senses.
2. Impairment of visibility and sky darkening.
3. Impairment to health of humans and animals.
4. Dark or light soiling.
5. Damage to vegetation.
6. Damage to materials.

Most discussion of air pollution tends to emphasize one of the first four of these effects because they are the manifestations with which people are most closely associated. Yet, the deterioration of inanimate materials resulting from atmospheric pollution is a significant drain on the economy of any heavily populated and industrial area. Although truly valid cost data are still not available, as early as 1930 it was estimatted that yearly damage to building property alone in England was in excess of two million pounds.[1] Much of this type of deterioration goes relatively unnoticed because it cannot always be distinguished from what might be called "normal" or "natural" deterioration.

The atmosphere which surrounds the earth is several hundred miles deep, but even so can be considered only the skin on the orange which is our globe. The thickness of the atmosphere which concerns us in air pollution problems is smaller by several orders of magnitude and can vary between a few thousand and a few hundred feet above ground, depending upon the dispersing properties of the atmosphere.

The atmosphere is mostly nitrogen with about 20 percent oxygen, about 0.03 percent CO_2 and the rare gases (A, Ne, Kr, He) and hydrogen making up less than 0.1 percent. The more abundant of these so-called rare gases exist in amounts which are several orders of magnitude greater than the concentration of some air pollutants which even at these infinitesimal levels can have devastating effects upon materials.

It has sometimes been implied that the only air pollutants which can effect materials are gases. This is not the case. In general, air pollutants can exist in any one of the following physical forms: 1. Solids; a) Large dust. b) Microscopic dust. 2. Liquids; a) Large droplets. b) Microscope droplets. 3. Gases.

In common with air pollution's undesirable effects otherwise deterioration of materials is not necessarily associated with any one of these physical forms. Interactions between physical forms, as well as atmospheric chemical reactions, have been found to be astonishingly complex.

Effect of Weather

The bulk of this discussion will deal with the effect of fairly specific atmospheric pollutants on a number of materials, but there is need to understand some of the physical factors which bring about the build-up of pollutants in the atmosphere and which cause them to come in contact and react with these materials. The atmosphere which bathes us is continually in motion, but the character of this motion varies over a wide range.

The simplest types of air pollution situations involve single point sources emitting single pollutants that cause deterioration of materials in the immediate surroundings. In such cases the pollutants producing the effect usually have not undergone any appreciable physical or chemical change between the time of their release and their contact with the affected materials. Therefore, it usually is possible to establish the cause and effect relationship by means of measurements of single pollutant concentrations as they relate to wind direction and

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velocity. In fact, given a point source of pollutant represented by a tall smoke stack free from obstruction, such as buildings, and located in level terrain, it is possible to predict (with various degrees of accuracy) the concentration at any point downwind of the stack under a variety of conditions of turbulence. Because localized situations such as these are fairly easy to recognize and only one source is involved, the means for correction should be straightforward.

It is the area-wide effects of air pollution from a multitude of sources of various kinds which require detailed study and is the justification for regional air pollution control agencies. In this type of situation the cause and effect relationship is not easy to establish.

Topography and the Temperature Inversion

Most heavily populated areas in hilly terrain are located in valleys because of the availability of waterways and other transportation arteries. From an air pollution standpoint this is undesirable because during periods of little wind movement pollutants tend to build up within these valleys.

These periods of low wind speeds often are associated with an atmospheric condition known as the temperature inversion which means that the temperature structure has become "inverted" from the so-called "normal" condition. To illustrate: If one were to measure temperatures vertically above ground over a level field somewhere in the Midwest on a bright sunny afternoon in late summer he would find that temperatures would probably decrease in a fairly regular manner with height. In the example, this would be considered a "normal" condition.

Along the West Coast, on this same afternoon say, in Los Angeles or Oakland, one might very well find that the temperature structure above ground would be quite different from this "normal" condition. Starting at ground level the temperature might initially decrease with height to a point several hundred feet above ground, then suddenly the temperature would begin increasing with height to some altitude. This would be called the inversion layer whose thickness and height above ground can vary considerably.

Under a simple "normal" condition, a puff of smoke or other pollutant would tend to rise and disperse continually because of the presence of turbulence and mixing in the normal air layer. In the "inversion" situation the pollutant would disperse until it reached the bottom part of the inversion layer where further dispersion would be prevented because of the lack of mixing or turbulent currents in the inversion layer. The inversion along the Pacific Coast is associated with the over-all weather conditions in this area and for hundreds of miles at sea and persists through the late summer and fall.

In most areas inversions are observed frequently on mornings following clear nights. They are called radiation inversions. This terminology is used to describe conditions which occur after the sun goes down and the ground begins to loose heat, thereby cooling the layer of air close to the ground. This layer of cooler air deepens through the night from continued radiation and produces the inverted temperature profile. On the floor of a valley the deepening of the inversion is accelerated because of cool air draining off of the hills into the valley. Pollutants emitted within this inversion layer tend to stratify without dispersing upward because of the poor mixing conditions.

In addition, without the warmth of the sun, the ground and exposed objects tend to become cooler than the surrounding air, and often their surface temperatures decrease to below the dew point. The resulting condensed surface moisture on materials in the presence of corrosive pollutants, whose concentrations are increasing under the stable influence of the temperature inversion, provide an ideal environment for the promotion of certain types of damage to materials.

Atmospheric Pollutants and Their Effects on Materials

In surveying the literature on atmospheric corrosion of metals,[2, 3] most data presented showed the rate of corrosion associated with the general types of atmospheres such as marine, rural, industrial, etc. Industrial categories imply the presence of a mixture of several atmospheric contaminants, but data were difficult to find in the corrosion literature on the exact chemical composition of these industrial atmospheres and the specific mechanism of attack. The emphasis of this paper is on the air pollutants; therefore the remainder of this discussion will deal with a group of fairly specific air pollutants known to produce deterioration of materials, will list the principal sources of these pollutants, and will discuss the most likely mechanisms by which deterioration of a variety of materials can occur.

Carbon Dioxide

This gas usually is not considered an air pollutant because it is a necessary ingredient of even the purest atmosphere for the building of plant tissues in the process of photosynthesis. Although the CO_2 content of the atmosphere is controlled by absorption in the oceans, rain and by vegetation intake, concentrations over heavily industrialized areas, where large amounts of fossil fuels are being consumed, can reach many times the so-called normal value of 0.03 percent.

Its principal undesirable effect can be found in the deterioration of building stones, and in particular such carbonate rock as limestone.[4] In the presence of moisture the resulting carbonate acid attacks calcium carbonate, converting it to the water soluble bicarbonate which is then leached away. It is doubtful that this type of deterioration would occur, as is the case with many other instances of atmospheric deterioration or corrosion, if it were not for the presence of moisture.

Sulfur Dioxide and Trioxide

Sulfur dioxide has its principal sources in combustion processes where fuels such as coal and oil containing appreciable amounts of sulfur (1 to 5 percent) are burned. From sources of this type in New York City it has been estimated that over 1.5 million tons of SO_2 are emitted per year.[5]

Other important sources of SO_2 are in the roasting of sulfide ores and refinery operations where sludges and waste gases high in sulfur are burned. Concentrations of this pollutant sometimes reach several ppm (parts per million) over short periods of time in areas immediately surrounding such large sources, but over extended periods in areas where there are many smaller sources of SO_2 the concentrations usually average less than one ppm. Even at these seemingly low levels, SO_2 is capable of damaging a wide variety of materials. As early as 1939 Burdick and Barkley[6] of the United States Bureau of Mines surveyed the available literature covering the deterioration of many materials in atmospheres containing SO_2. Much of the work was carried out in laboratories under conditions of concentration and pressure which would not normally exist in the free atmosphere, but nevertheless a wide variety of materials was shown to be affected.

Sulfur trioxide is likely to be formed whenever SO_2 is formed in a process, but in much lesser amounts.[7] Because of its extremely hygroscopic nature it invariably exists in the atmosphere in droplets of sulfuric acid. SO_2 in the presence of sunlight is capable of undergoing auto-oxidation to SO_3 in the atmosphere. The rate for this reaction is low (0.1 to 0.2 percent per hour) if the air contains no other contaminants, but there is increasing evidence that certain constituents in particulate matter are found in most polluted atmospheres greatly increases this reaction rate. Further reactions of the acid with other air pollutants appear to result in the formation of sulfates.

Metals. Although aluminum appears to resist attack of sulfur dioxide,[8] many other metals (particularly iron and steel) are strongly susceptible to attack. Those metals which are attacked by SO_2 and SO_3 require the presence of water in vapor or liquid form for the attack to occur. The rate at which iron rusts is greatly accelerated by traces of SO_2 in the air. However, in bone dry air SO_2 has little or no effect on iron and many other metals. It appears that the combination of moisture, SO_2, and particulate matter combine to promote attack on iron.

In work carried on at the Chemical Research Laboratory in Teddington, England,[1] it was shown rusting in a moist atmosphere containing SO_2 is greatly accelerated by the presence of particulate matter. In an experiment, one sample of iron was exposed to a moist atmosphere containing traces of SO_2. Another sample was exposed to the same atmosphere, but protected from particu-

late matter by means of a muslin cage which permitted only the gaseous constituents to contact the sample. The rusting of the protected sample was negligible compared with the sample which was unprotected. It was not known how much of the moisture and SO_2 were absorbed by the fabric making up the cage, or whether equilibrium was established.

Sulfur oxides enter into the formation of the well known greenish coating on copper and copper alloys in industrial areas. This consists of basic copper sulfate,[9] which is extremely resistant to further atmospheric attack and therefore acts as a protective film. However desirable this effect may be from the standpoint of esthetics and further attack, these same reaction products are unwanted when they form on electrical contacts made of copper and thereby change the electrical resistance of the contacts.

Building Materials. SO_2 is capable of attacking a wide variety of building materials, including limestone, marble, roofing slate, and mortar. Any carbonate-containing stone is damaged by having the carbonate converted to relatively soluble sulfates which can be leached away by rain water. Other types of stones, such as granite and certain sandstones, in which the grains are cemented together with materials containing no carbonate, are relatively unaffected by SO_2 in the atmosphere.[10]

Leather. SO_2 has a strong affinity for leather. This pollutant causes leather to lose much of its strength and ultimately to disintegrate. As early as 1843 Faraday came to the conclusion that the rotting of leather upholstery on chairs in a London club was the direct result of sulfur compounds in the air.[11]

The storage of valuable leather bound books in city libraries can pose a serious problem. The bindings of books stored in rooms with a free exchange of polluted air were found to deteriorate much more rapidly than those stored in confined spaces or inside of glass cases.

Leather safety belts exposed to polluted atmospheres containing SO_2 can be dangerously weakened in a few years' exposure. The analysis of leather which has rotted under these conditions show sulfuric acid contents as high as 6 or 7 percent.

Paper. In Burdick and Barkeley's Review[6] a report of the work of one researcher[12] showed that SO_2 in concentrations of 2 to 9 ppm for a 10-day exposure resulted in embrittlement and a decrease in folding resistance of both book and writing papers.

Textiles. In many areas the average person thinks of air pollution in terms of the dirtying of textiles, yet sulfur oxides alone are capable of causing deterioration to a variety of natural as well as synthetic fibers used in textiles.

Cotton, being a cellulosic fiber like paper is weakened by the presence of SO_2. The work of Race[13] in England as reported by Greathouse[4] showed that the breaking strength of cotton fabric in winter was considerably less than that found in summer. Without atmospheric pollutants one would expect the reverse to be the case because of the higher sunlight intensity in summer which in itself can cause deterioration of the fabric.

Wool, being a proteinaceous material behaves like leather and is capable of absorbing and being weakened by SO_2.

Damage to women's nylon hose by air pollution (presumably SO_2 or SO_3) has made newspaper headlines on numerous occasions in many areas. The exact mechanism of the attack has not yet been thoroughly investigated and reported, but it has been shown that a 3 percent solution of sulfuric acid is capable of reducing the strength of nylon (apolymeric amide) yarn by about 20 percent.[14] It has been postulated that extremely small atmospheric particles containing adsorbed SO_2[15], or tiny droplets of sulfuric acid which have formed around particles[5] become attached to the very thin nylon fibers and these, being under some tension, fail. Other materials of nylon, such as the fabric in shirts and dresses, are made from fibers of much larger diameter than those of nylon hose and are not under the same tension and apparently are not as susceptible noticeably to this kind of damage.

Hydrogen Sulfide

This gas is often associated with the malodor of rotten eggs. There is some question of its odor threshold, but it is probably below one ppm.

One of its principal sources has been refinery operations where "sour" crude oil is processed. However, because the sulfur can be recovered from this gas in a process that in some instances ultimately may pay for itself, H_2S from this source is decreasing.

Also H_2O is released from bodies of water into which substantial amounts of inadequately treated sewage have been dumped. Because of the oxygen-deficient environment, the normal sulfate content of these waters is reduced to sulfide, which can be released as H_2S. Another source of H_2S is in the manufacture of coke from coal containing appreciable sulfur.

Hydrogen sulfide can be oxidized in the atmosphere to SO_2 and SO_3, particularly under conditions of high humidity and to some extent its responsibility in atmospheric deterioration can be related to the preceding section on sulfur oxides. However, there are several forms of deterioration which are related directly to hydrogen sulfide gas.

Paint. House paint containing lead compounds is rapidly darkened in the presence of even low concentrations of H_2S by the formation of black lead sulfide. The work of Hess[16] as reported by Greathouse[4] points out, however, that the dark lead sulfide is oxidized rapidly to lead sulfate, thus turning the paint film white. It has been observed in Detroit that pigments in a lead base paint exposed to moist H_2S may show a different color, such as pink or tan.

Because highly polluted atmospheres often contain appreciable quantities of dark particulate matter there is the chance that the dirtying of a painted surface by this kind of material can be mistaken for lead sulfide darkening.

Another type of paint surface darkening which has been confused with hydrogen sulfide damage is caused by certain types of fungus.[17] This kind of damage occurs most frequently where the humidity remains above 60 percent. It is likely to occur on paints containing a high percentage of raw linseed oil and some of the newer latex emulsion based exterior paints.

Metals. In the presence of H_2S, such metals as silver and copper tarnish rapidly. The sulfide coating formed on copper and silver electrical contacts when separated can increase greatly the resistance across these contacts when they are closed. This also may result in welding the contacts.

Hydrogen Fluoride

This extremely dangerous and corrosive gas is evolved from a variety of processes which include the following:

The production of synthetic fertilizer starting with the acidulation of phosphate rock which invariably contains combined fluorides.

The manufacture of enamel frits and high fluoride glass.

The production of basic aluminum which uses cryolite and other fluorine compounds in the electrolytic reaction.

The production of steel where the ore contains fluorides and where fluorspar is used as a flux.

Being a corrosive gas and at the same time extremely hygroscopic, it can attack a wide variety of metals and glass, or enamel-coated materials.

There is at least one report in the literature[18] of HF attacking window glass to the extent that the glass was rendered opaque. This effect also has been noted by the author on several occasions. It should be pointed out, however, that the concentrations required to produce visible damage to glass and other materials are felt to be far in excess of those required to kill many types of vegetation.

Certain types of plants such as gladiolus are extremely sensitive to HF and are damaged by concentrations in the parts per billion dilution range. In addition, cattle feeding on vegetation exposed to relatively low concentrations of HF are subject to fluorosis. Therefore, other than occasional localized damage, hydrogen fluoride does not appear to be an important contributor to the deterioration of materials simply because it cannot be tolerated for other and more urgent reasons.

Ozone and Photo-Chemical Smog

Ozone has been measured at various points over the world and concentrations at ground level in most areas appear to be in the range between 0 and 5 parts

per hundred million (pphm). Within the stratosphere the concentrations may be as high as 600 to 800 pphm. However, it is unlikely that the low natural levels of ozone near the ground result from diffusion downward from the stratosphere. The natural ozone concentrations at ground level are more likely to be formed by the action of sunlight. Regardless of this, ozone levels in certain polluted atmosphere such as those found under so-called "smog" conditions in the Los Angeles area and, to a somewhat lesser degree, in the San Francisco Bay Area, can rise to concentrations many times those associated with normal "background" levels.

The relatively high ozone levels are related to a series of complex chemical reactions occurring in the atmosphere,[19] many of which are now understood, but many of which are still being learned about. Haagen-Smit[20, 21] and his coworkers carried out the first experiments which showed that low concentrations of hydrocarbons in the presence of nitrogen oxides and sunlight produced unusually high ozone levels. Without sunlight the ozone levels drop back to the "normal" range. More recent work at the Franklin Institute[22] has contributed considerable insight into the nature of these reactions but, at the same time, has confirmed earlier observations that the chemistry of polluted atmospheres is fantastically complex.

The principal sources of photo-reactive hydrocarbons and other organic vapors in the atmosphere are the exhausts from automobiles and other internal combustion engines, refinery operations, solvent losses from a variety of operations and inadequate incineration. Oxides of nitrogen are part of the normal effluent from any high temperature combustion operation in which air is the source of oxygen, because some of the nitrogen is oxidized.

The high ozone level during some smog conditions is but one of the undesirable manifestation; others include eye irritation, reduced visibility from the formation of aerosols, and vegetation damage. However, the high ozone content or oxidizing capacity of atmospheres polluted in this manner is largely responsible for most of the observed serious deterioration to materials associated with this type of smog.

Rubber and Elastomers. One of the early indications of the smog problem in Los Angeles was the excessive cracking of rubber products. It had long been known that atmospheric oxygen and especially ozone could cause rubber cracking. Crabtree and Kemp[23, 24] carried out extensive studies as early as 1946 on the attack of natural rubber by light-energized oxygen and ozone. The cracking of rubber in an ozone atmosphere of even high (1 to 5 percent) concentrations does not appear to proceed until the rubber is stretched. Even the slightest stretching under these conditions brings about rapid deterioration.

The Los Angeles County Air Pollution Control District uses as one of its measurements of ozone concentration in the atmosphere a method developed by Bradley and Haagen-Smit[25] in which bent strips or rubber of precise formulation are exposed to the air to be sampled. By determining the depths of cracks at the end of definite exposures it is possible to estimate the ozone content of the atmosphere.

According to present theory, elastomers of the unsaturated type are attacked by ozone at the double bond in the carbon-to-carbon chain.[26] If the chain is under stress, it breaks, which leaves its neighbor under additional strain. Synthetic rubbers such as butadiene-styrene, or butadiene-acrylonitrile, being unsaturated, are affected in this same manner. Neoprene, although unsaturated, resists ozone attack, presumably because of the presence of the chlorine atom adjacent to the double bond. Butyl, polysulfide and silicone polymers, being saturated, are highly resistant to attack by ozone.

Literature references on the subject of deterioration of rubber in ozone-containing atmospheres testify to the seriousness of this kind of damage. This effect is especially important relating to tires and insulated utility wires.

Textiles and Dyes. Ozone has long been known to affect the strength of fabrics and the color fastness of dyes.[27, 28, 29, 30] Only recently, however, have any experiments been caried out to determine the quantitative effects of ozone as related to community air pollution mixtures on fabrics and dyes. Some recent research by a commitee of the American Association of Textile Chemists and Colorists[31] presented preliminary results on the exposure of a group of different fabrics and dyes to Los Angeles smog. The samples were exposed in chambers which provided free passage of contaminated air inside the chamber, but permitted no direct sunlight to strike any of the samples. Because this was a preliminary investigation, no conclusions were drawn other than the statement that exposure to smog conditions did cause definite deterioration of a number of fabrics and dyes.

It is certain that this type of smog is detrimental to a number of natural and synthetic materials, but the awareness of and understanding of the many factors is so new that active research projects on several of the factors are just now getting underway.

Solid Particulates and Other Damaging Agents

The role of solid air pollutants in causing deterioration normally is thought to be that of soiling. This can be either the soiling of light surfaces such as a table cloth, or white marble column on a building, with dark-colored particulate matter such as soot; or the soiling of a dark surface, like a polished mahogany table by light colored solids such as cement dust. The soiling does not necessarily cause deterioration directly, but its removal does. Much money has been spent in cities where large quantities of coal are burned, to sandblast off the sooty layers which have accumulated on prominent buildings. One aspect of this effect stressed by Greenburg and Jacobs[5] is somewhat unique; the tarry components of carbonaceous material resulting from poor combustion of coal or oil are likely to be sticky and also acidic in nature. Therefore, their corrosiveness is likely to persist over extended periods when they are not flushed off by rain.

The direct or chemical deterioration of materials as a result of solid particulates collecting upon their surfaces depends upon the chemical activity of the solid in its environment and the susceptibility of the receiving material.

Preston[32] "inoculated" various metallic surfaces (coated and uncoated) with fine powders or "nuclei". He used such materials as sodium chloride, ammonium sulfate and chloride, sodium nitrate and flue dust. The samples were then exposed to atmospheres held at various humidities and the resulting corrosion was measured. The reaction was the typical filiform corrosion observed from many highly reactive particulates in the atmosphere. In most of the cases in this study corrosion increased with humidity. One exception was ammonium chloride. However, the addition of traces of SO_2, to the test atmosphere greatly increased the corrosion at high humidities. It should be pointed out that these experiments were carried out in a laboratory environment and did not duplicate actual outdoor conditions.

A more specific occurrence of damage to materials from atmospheric solids resulted from the deposit of iron particles from a grinding operation on parked automobiles.[33] It was noted that brown particles were attaching themselves tenaciously to the paint of automobiles in a parking lot. A brown stain surrounded each particle and many cars had to be repainted. The particles at the center of each stain were identified microscopically as iron grindings or cuttings based largely on their peculiar shape. The mechanism of the staining was felt to be more complex than simple rusting. The authors postulated that it resulted from the formation of ferrous hydroxide in the presence of moisture which, being in a colloidal form, diffused into the paint film, and upon drying and oxidizing left the brown stain of ferric oxide.

While discussing damage to automobile finishes it should be pointed out that the automobile is an excellent indicator of corrosive or damaging atmospheres because the owner always has extreme pride and large investment in ownership and most instances of damage are quickly brought to the attention of those in charge of air pollution control programs. In Detroit[34] the air pollution control agency accumulated numerous records of various kinds of damage to automobile finishes.

The same type of damage from iron particles as mentioned above was experienced in Detroit. However, it was theorized there that in the presence of moisture some of the oxygen for the formation of iron oxide came from the paint itself. The result was a pit, filled and over-covered with a bump of iron oxide.

Cars parked near demolition opera-

tions where brick buildings were being taken down were severely damaged by mortar dust in the presence of moisture. The damage could be reproduced by sprinkling mortar dust on paint test panels and covering with a moist blotter. The damage had the appearance of crazing. No attempt was made to identify the damaging agents in the dust.

Other damage to auto finishes in Detroit was by chromic acid mist, which formed a brown stain on light-colored cars, and a "blush" on the darker shades of paint. Repainting was required because the color change could not be removed by washing or polishing.

Sodium carbonate particles from such operations as soda ash manufacture, or as a breakdown product from cyanide heat-treat bath evaporation created a characteristic pit in the paint surface. Each pit contained a white particle and the pits were thus easily identifiable on dark paint surfaces. The white specks could be removed by washing, but of course the damaging pit remained.

Conclusions

The deterioration of materials resulting from atmospheric pollution is a serious economic loss. Beyond this, it is related to a number of complex atmospheric and surface reactions, all of which could use further study. Atmospheric corrosion engineers and specialists who must investigate deterioration of materials need to familiarize themselves thoroughly with the physical environment in which they carry out their measurements. This knowledge of the environment includes not only the physical factors which influence the form and manner in which the agent contacts the materials, but perhaps more important knowledge of the chemistry of the specific agents which singly (or in combination) lead to the ultimate damage to materials.

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Any discussion of this article not published above will appear in the December, 1959 issue.

NACE Guide for Preparation of Articles for Publication

Persons interested in submitting articles on corrosion for publication in CORROSION can obtain upon request a copy of the "NACE Guide for Preparation of Papers." Write to CORROSION, National Association of Corrosion Engineers, 1061 M & M Bldg., Houston 2, Texas.

Recent Metals Corrosion Articles Published in CORROSION

Design Against Atmospheric Corrosion by Henry T. Rudolf. *Corrosion,* **11,** No. 8, 347t-350t (1955) Aug.

Specific recommendations for construction of metallic surfaces exposed to corrosion are made. The author points out total exposed areas may be reduced by adopting the proper shapes of structural members, by enclosing with planar plates structures including angles and braces. Seal welding is recommended.

The design engineer is warned to keep in mind structures exposed to atmosphere ultimately require surface preparation and that an effort should be made to minimize inaccessible areas. Consideration should be given to the need for ultimate painting by providing as many plane surfaces as possible. Welding is favored over riveting. Specific recommendations are made concerning design which offers the best possible way of minimizing corrosion damage.

Atmospheric Galvanic Couple Corrosion by K. G. Compton, A. Mendizza and W. W. Bradley. *Corrosion,* **11,** No. 9, 383t-392t (Sept.) 1955.

Tests designed to provide fundamental information explaining the behavior of galvanic couples in the atmosphere are reported. The purpose is to permit prediction of their probable relative behavior. Measurements of weight losses of couples in a marine, an industrial and a severe tropical atmosphere are reported.

Tests involving thin sheet electrode material separated by filter paper saturated with electrolyte are reported. Materials tested include aluminum, magnesium, stainless steel, copper, cadmium, zinc, nickel, tin, chromium, lead, electroplate, silver, low carbon steel and others. Potential measurements and weight loss data are given. Specimens exposed to the atmospheres show several apparent weight loss anomalies.

Measurements of corrosion currents described provide a quick means for determining probable relative corrosion behavior of couples. Differences between couples "exposed" and "sheltered" are noted. Quantitative data provided are intended to be useful to the design engineer.

Corrosion Testing Farm at Mandapam Camp by K. S. Rajagopalan, M. Sundaram and P. L. Annamalai, *Corrosion.* December, 1959.

Monthly rates of corrosion of mild steel, zinc and copper at two sites located 150 feet and 1350 feet from the sea are reported. Coupons were exposed in under both open and sheltered conditions on racks oriented at 45 degrees facing south.

Climatic conditions at the Mandapam Camp site are discussed in detail and means of keeping the records reported. Rates of corrosion of the test samples are discussed in relation to the observed climatic condition. Differences between the open and sheltered tests are compared.

Among the conclusions: There is no direct relation between the corrosion rate, distance from the sea and atmospheric salinity, although salinity is an important factor in the rate. Highest rates were observed in months with high mean temperature and accompanying high average humidity and salinity. Corrosion rate does not vary in proportion to distance from the sea under sheltered conditions.

The Corrosion Behavior of Aluminum by Hugh P. Godard. *Corrosion,* **11,** No. 12, 542t-552t (1955) Dec.

The practical corrosion behavior of aluminum is reviewed for the non-specialist. Included is a discussion of the following topics: the relative nature of corrosion resistance, resistance criteria, factors influencing corrosion rates, common corrosion problems, how to choose an alloy; and galvanic, deposition, crevice and pitting corrosion. Specific information is given on corrosion in six major environments. Other topics considered include corrosion of brazed and welded joints, atmospheric performance of anodized aluminum and preventive measures.

Twenty-nine references are appended for persons interested in further study.

The Relation of Thin Films to Corrosion by Thor N. Rhodin. *Corrosion,* **12,** No. 9, 465t-475t (Sept.) 1956.

Recent studies of thin oxide films isolated from stainless steels and other ferrous alloys have provided new information on surface properties. Correlations between the unique compositional and structural properties of thin films and alloy corrosion behavior in various corrosive environments have been established. Evaluation of the resistance to pitting corrosion of silicon-modified Type 316L stainless steel is described as a typical example of an application of a film study to corrosion research. In this case, improved corrosion behavior was correlated with a mutual film-enrichment of silicon and molybdenum and a film-depletion of iron. Experimental alloys of improved purity were prepared and evaluated. Effects of alloy purity, carbon content, and alloying additions were found to influence strongly surface behavior and corrosion resistance.

The mechanism of resistance to pitting corrosion and the protected nature of the associated oxide films are interpreted in terms of the chemistry of hydrated heteropoly acids containing oxides of silicon and molybdenum.

Atmosphere Affects the Stress-Corrosion Failure of High Brass, by C. H. Hannon. *Corrosion,* **13,** No. 6, 417t-418t (June) 1957.

Observed failures of high brass parts after prolonged service as arcing gap electrodes in electrical apparatus led to a special laboratory investigation. Failures of the electrodes was evidenced by disintegration, embrittlement, cracking and obvious oxidation. This article reports the observations made covering the effects of ammonia, oxygen, nitrous oxide and ozone on highly stressed brass test pieces.

Corrosion of Metals in Tropical Environments. Part 1—Five Non-Ferrous Metals and a Structural Steel by B. W. Forgeson, C. R. Southwell, A. L. Alexander, H. W. Mundt and L. J. Thompson. *Corrosion,* **14,** No. 2, 73t-81t (Feb.) 1958.

An extensive investigation has been in progress since 1940 in the Panama Canal Zone to study the corrosion rates and characteristics of fifty different metals and alloys exposed to five natural tropical environments for periods of one, two, four, eight and sixteen years. Up to the present, specimens through the eight year period have been removed and examined. The report describes the corrosion rate of aluminum, lead, nickel, zinc and copper along with structural steel. Corrosion data for these metals as observed in the five environments have been obtained from weight loss and pitting measurements, visual inspection and physical property studies. Corrosion rates for these metals in the tropics are related to corrosion rates previously reported for these metals in temperature latitudes.

Corrosion of Metals in Tropical Environments. Part 2—Atmospheric Corrosion of Ten Structural Steels by C. R. Southwell, B. W. Forgeson and A. L. Alexander. *Corrosion,* **14,** No. 9, 435t-439t (Sept.) 1958.

Results are reported of 8-year exposure tests made to determine the corrosion characteristics of ten structural steels exposed to seashore and inland environments in the Panama Canal Zone. These corrosion rates are compared with existing data for the same materials in temperate zones.

Data reported include a summary of individual analyses of air samples obtained at atmospheric test sites and extensive weight-loss and pitting data for the ten steels at 1, 2, 4, and 8-year exposure intervals. Numerous time-corrosion curves in tropical marine and tropical inland exposures are given for the steels.

Considerable attention is given to the effect of low-alloy additions on mild carbon steel, It was found that the corrosion of copper steel in the two tropical atmospheres was only 13 percent and 14 percent less than that of the unalloyed mild steel. The 2 percent and 5 percent nickel steels were very effective in resisting corrosion as was nickel steel. The four proprietary low-alloy steels tested displayed good corrosion resistance to tropical atmospheric corrosion.

Corrosion Experience Associated With Hydrometallurgical Refining of Nickel At Sherritt Gordon Mines*

By R. B. McINTOSH*

Abstract

Some of the conclusions drawn regarding materials of construction for the Sherritt Gordon Ammonia Leach process during pilot plant study from 1949 to 1954, are compared with experience gained after four years of refinery operations. In addition, a few corrosion experiences are described in more detail and some mention is made of general corrosion problems. 7.6.9

Introduction

TO EXPLOIT its Lynn Lake nickel-copper ore-body, Sherritt Gordon Mines Limited has developed a novel hydrometallurgical process from small scale laboratory experiments to full scale refinery operation. In the spring of 1949, laboratory work had shown that the process was feasible and a pilot plant was operated in Ottawa until 1954. One important phase of the pilot plant program was the evaluation of materials of construction for various stages of the process.

The purpose of this article is to compare some of the conclusions drawn regarding materials of construction during pilot plant study with experience after four years of operation. In addition, a few interesting corrosion experiences will be described in more detail and some mention will be made of general corrosion problems.

Description of Process

Complete descriptions of the ammonia leach process used by Sheritt Gordon Mines can be found in several papers listed in the bibliography and, therefore, a brief outline only will be given here. A block flowsheet of the process is shown in Figure 1.

The sulfide concentrates produced by conventional flotation techniques from the ore at Lynn Lake, Manitoba, contain approximately (%) 12 nickel, 0.5 cobalt, 1.5 copper, 30 iron, and 28 sulfur.

At the refinery this finely divided material is repulped in an ammonia solution and pumped to leaching autoclaves. Under conditions of elevated temperature and pressure, oxygen, supplied from a continuous flow of compressed air, reacts with the sulfide minerals to produce a solution of ammonium sulfate containing the metals as complex ammine salts. Iron remains with the undissolved solids mainly as hydrated ferric oxide. The tailings are separated from the pregnant solution by a series of thickeners and filters designed to recover as much of this solution as possible. Ammonia is recovered from the spent leach gases as aqueous solution by a bubble-cap absorption column.

The ammonia in the leach solution in excess of that required for subsequent operations is removed by distillation and is recycled to the leaching circuit. Simultaneous precipitation of copper sulfide permits recovery of this metal in a form which can be shipped to a conventional smelter for refining. Complete removal of copper from the solution is effected by addition of controlled amounts of hydrogen sulfide.

Residual unsaturated sulfur compounds formed in the leaching step and not consumed in the copper precipitation, together with considerable amounts of ammonium sulfamate, are converted to sulfate in a combined oxidation and hydrolysis operation. Nickel is recovered from the purified solution by a batch reduction operation using hydrogen as the reducing agent and ferrous sulfate as the nucleation catalyst. The nickel powder produced in this operation is washed, dried, briquetted, sintered and packaged. The unreduced nickel, about 1 gm/liter, together with the cobalt extracted from the concentrate, is recovered from the reduced solution by precipitation with hydrogen sulfide for subsequent recovery of the cobalt. The metal-free solution is evaporated to crystallize and recover fertilizer grade ammonium sulfate.

Raw material for synthesis of ammonia is obtained by reacting natural gas with steam and air and scrubbing the resulting nitrogen and hydrogen with monoethanolamine, copper ammonium formate and caustic to remove carbon dioxide, monoxide and other impurities. The nitrogen-hydrogen mixture is passed through a standard converter to produce ammonia.

Pilot Plant Conclusions Versus Refinery Experience

Leaching

A more detailed schematic flow diagram of the leach and filtration circuits is shown in Figure 2. Solutions throughout the circuit are strongly basic with considerable excess ammonia. The first stage solutions contain higher concentrations of nickel, copper, cobalt and sulfur than the second stage ones, but they do not differ a great deal in their corrosive nature. However, solids in the first stage slurries have not been leached as much as those in the second stage, so they contain more sulfidic material. This difference between first and second stage slurries is the prime factor in any difference that has been experienced in the corrosive characteristics of these slurries.

During pilot plant study, two main corrosion reports were issued, one in February of 1952 and the other in September, 1953. Table 1. shows the results from each report for the first stage leaching autoclave.

Considerable differences between these two tests can be seen. Mild steel corrosion is considerably higher in the later test while that of all the stainless steels is considerably less. However, after the first test it was noted that specimens were loose on the holder and some erosion had taken place around the holes. It was suggested also that some contact corrosion had taken place under spacers.

Unfortunately, equipment for the refinery had to be ordered before complete results were available from the second

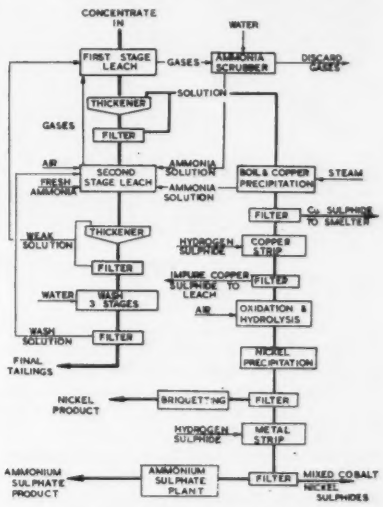

Figure 1—Metals plant flowsheet

TABLE 1—Pilot Plant Corrosion Data
First Stage Leaching Autoclave

Date	February, 1952		September, 1953	
Duration of Test	30 days		107 days	
	Corrosion Rate in/yr		Corrosion Rate in/yr	
Materials	Annealed	Sensitized	Annealed	Sensitized
Mild Steel	.006	....	.025	.....
Type 304 S S	.001	.001	.00002	.00005
Type 316 S S	.001	.002	.00004	.00007
Type 316L S S	....	....	.00004	.00004
Type 347 S S	.001	.002	.00002	.00004

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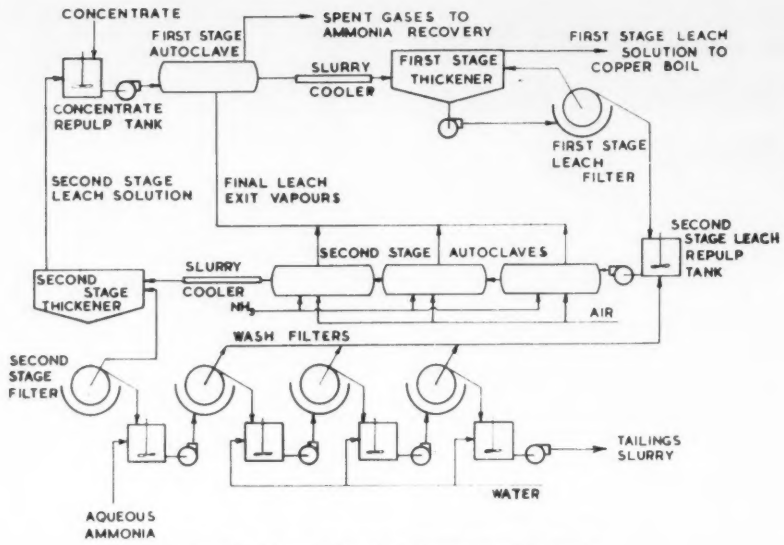

Figure 2—Schematic flow of leaching and filtration circuits.

Figure 3—Corrosion in carbon steel experimental filter.

test. A total of eight leaching autoclaves, 11 ft in diameter by 45 ft long were ordered as carbon steel clad with Type 316 extra low carbon stainless steel. All internal parts were either Type 316 or 316 ELC stainless steel.

Even if results had been available from the second test, it was probably good insurance to have the autoclaves constructed of Type 316 ELC clad instead of 304 clad because the extra material cost would not be very significant in relation to the overall cost of fabricating and installing the autoclaves.

Most of the other less costly items of equipment such as filters, storage tanks, piping, etc., were ordered in Type 304 or 304 ELC material.

Refinery experience has verified corrosion test results from the second test period in that Type 316 stainless steel is not needed anywhere in the leaching circuit.

Experience with mild steel in this circuit is not so clear cut. Steel is being used satisfactorily in many applications such as cooling coils in some autoclaves, shafts and impellers in all autoclaves and as shafts and impellers in many repulp tanks.

However, fairly rapid corrosion of carbon steel has been experienced on some autoclave cooling coils and on certain parts of one filter. It has proved quite difficult to predict, prior to actual trials, whether or not carbon steel would be satisfactory for different purposes in this circuit. Some examples to illustrate this difficulty can be cited.

An experimental carbon steel filter with slowly moving paddles in the filter boot was examined after about eight months of operation and severe metal loss calculated at .284 in/yr was noted on the filter boot. Areas of metal loss coincided with the location of the paddles as shown in Figure 3.

Evidence thus pointed to an erosion-corrosion condition. However, there was very little metal loss on the paddles. Elsewhere in some repulp tanks and leach autoclaves, where carbon steel is being used for shafts and impellers, the erosion-corrosion conditions should be quite severe. In many of these applications, carbon steel is performing quite well.

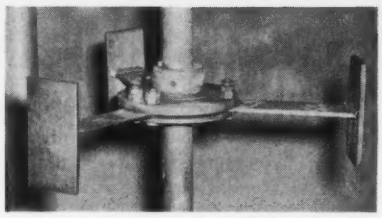

Figure 4—Typical design of original leach impeller.

Over the years, as production was increased, more cooling was required in the leaching autoclaves and many carbon steel coils were installed. Performance of these coils also illustrates the difficulties that arose in predicting the life of carbon steel parts. Coils were first installed in two compartments of each of the two first stage leach autoclaves which operated in parallel under identical conditions. During the first three and one-half months of its operation, one coil had a maximum pitting rate equivalent to .515 in/yr but during the next four and one-half months the rate decreased to less than .100 in/yr. In the other autoclave the pitting rates for an equivalent coil were .103 in/yr for the same three and one-half months period and about .510 in/yr for the next two months. It then operated for two years with a relatively mild pitting rate of less than .050 in/yr but then suddenly, over the following three months period the rate jumped up to about .58 in/yr.

Factors believed to have some bearing on this erratic behaviour were agitation, concentrate feed rate and type of concentrate. Pitting apparently was initiated by sulfidic material depositing on the coils. If agitation was such that the coils were swept clean, then very little pitting occurred. The nature of the concentrate seemed to determine the severity of pitting under these deposits. Changes in concentrate characteristics could possibly change the concentration and nature of reducing compounds such as $S_2O_3=$, $S_4O_6=$, and H_2S which can form under the deposits and thus change the activity of concentration and oxygen corrosion cells. In the second stage leach, even though there is normally some deposit on the coils, corrosion is not severe. However, if there is a dead space where solids never move, corrosion occurs at an accelerated rate.

It is virtually impossible to make a reasonable comparison between pilot plant corrosion test results on carbon steel and refinery experience. Test work showed that the corrosion rate would be 0.025 in/yr with some pitting evident while refinery corrosion has varied from practically nil to excessive pitting rates as previously discussed. This shows that test specimens cannot always be relied on to give completely accurate results and that when a material is sub-

Figure 5—Erosion of leach impeller blades.

ject to any pitting in small scale tests it should be used with caution for large scale equipment where slight changes in conditions may or may not accelerate this pitting.

During the pilot plant operation, considerable effort was expended in evaluating different materials and hard surfacing for impellers of the design shown in Figure 4. The original stainless steel leach autoclave impellers in the refinery were the same design and larger in all dimensions. It is interesting to note that the erosion rate on first stage leach blades increased by seven times in the refinery over that recorded in the pilot plant. Tip speed of refinery impellers was somewhat higher at 1950 ft/min than at 1780 ft/min in the pilot plant. Eroded blades are shown in Figure 5. All impellers of this type were replaced with swept-back turbine types made from carbon steel with hard-faced tips. These turbine impellers outlast the older type by two to three times before maintenance is required and they are much less expensive.

Copper Removal

In the copper removal circuit as shown in Figure 6, first stage leach solution passes through a series of four pots into a reboiler. Ammonia is distilled from the solution at about 220 F and condensed; while, in the reboiler and last pot, copper sulfide is precipitated. Filtered solution from the reboiler is treated with hydrogen sulfide to remove the remaining copper as copper-nickel sulfide which is recycled to the leach circuit. Although the solution loses most of its excess ammonia during this treatment, it still remains somewhat alkaline because nickel is present as the diammine complex. The concentrations of nickel and ammonium sulfate are increased.

Both pilot plant corrosion reports indicated that annealed Type 304 and 316 stainless steel were satisfactory but that in the sensitized condition only 316 ELC stainless steel remained relatively unattacked in this service.

Therefore, practically all equipment was constructed of Type 316 ELC stainless steel. Where copper sulfide is present the equipment generally has a hard dense coating of sulfide which does not appear to produce crevice type of attack. Since there is some light etching of the stainless steel in the reboiler, it would seem that 316 material was justified here but in the pots there is no sign of attack so presumably a less resistant

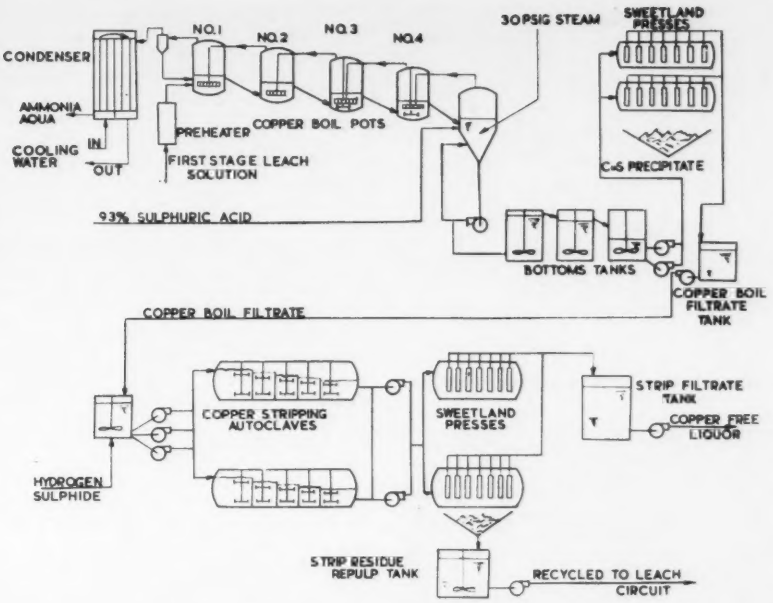

Figure 6—Schematic flow diagram of copper removal circuit.

austenitic stainless could have been used. The Sweetland filter presses were lined with thin gauge 316 ELC and have given some trouble with leaking welds. However, because there is no appreciable corrosion, this trouble probably is mechanical in nature, caused by differential contraction and expansion of the stainless steel and cast iron backing materials.

Very little opportunity has been presented for trying different materials in this circuit since few changes have been made. However, miscellaneous items such as filter chutes, pipelines, etc., have been installed in Type 304 stainless steel with no sign of failure.

A carbon steel shell and tube heat exchanger was installed originally to condense ammonia and water vapor from the No. 1 pot. No trouble was expected from this unit but carry-over of solution and copper sulfide with ammonia was not expected. In practice it was found that carbon steel tubes were perforated at the liquid-vapor interface. These tubes were replaced in Type 304 stainless steel. Since that time, operation of the copper boil unit has been very steady with no carry-over and under these conditions the original carbon steel tubes would undoubtedly have been satisfactory.

Nickel Recovery

Copper-free liquor containing nickel, cobalt, ammonium sulfate and unsaturated sulfur compounds is oxidized with air at 350 F and hydrolyzed at 450 F to convert all the unsaturated sulfur compounds and ammonium sulfamate to sulfate. It is then fed to a reduction feed tank and to the reduction autoclaves as shown in Figure 7. After the reduction of nickel at 350 F with hydrogen is complete, powder and reduced solution are discharged to flash tanks where the powder settles and is discharged periodically to be filtered, washed, dried, bri-

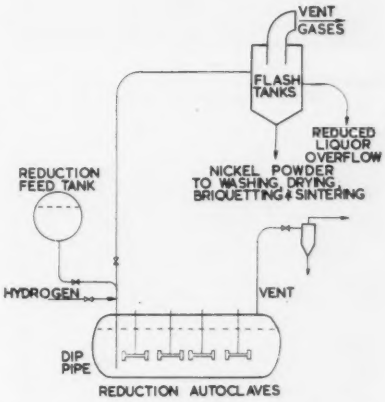

Figure 7—Schematic flow diagram of nickel reduction circuit.

quetted and sintered. Reduced solution, containing the cobalt and residual nickel, is treated with hydrogen sulfide at 200 F to precipitate a cobalt-nickel sulfide which subsequently is leached in acid solution with air, producing a solution from which nickel and cobalt can be recovered separately or as a mixed metal. Metal-free solution is evaporated to produce fertilizer grade ammonium sulfate.

Reduction of nickel ions with hydrogen produces nickel metal and hydrogen ion which reacts with the ammonia released from the diammine complex ion. After reduction, therefore, the solution is nearly neutral with a pH of 6.5 to 7.5 and contains a high concentration of ammonium sulfate.

Pilot plant corrosion tests were not complete before equipment was ordered for this section of the refinery and practically all material in contact with these solutions is either Type 316 or 316 ELC stainless steel. Subsequent results of pilot

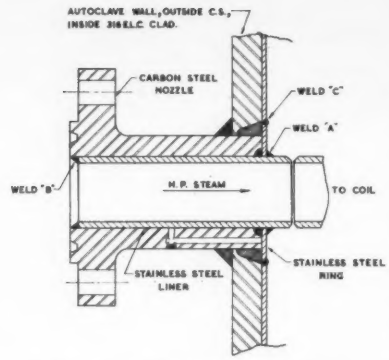

Figure 8—Steam nozzle on reduction autoclave.

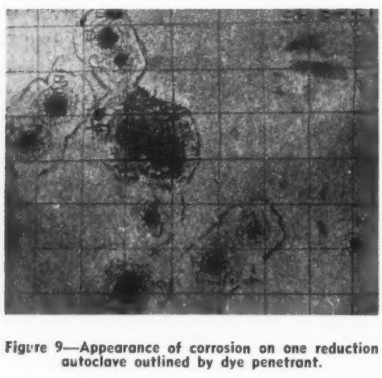

Figure 9—Appearance of corrosion on one reduction autoclave outlined by dye penetrant.

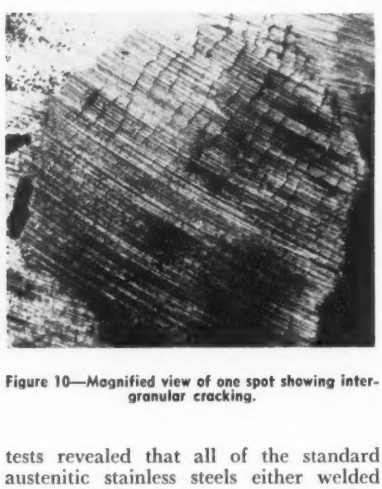

Figure 10—Magnified view of one spot showing intergranular cracking.

tests revealed that all of the standard austenitic stainless steels either welded or unwelded had corrosion rates of less than 0.0005 in/yr in a 25-day test.

Refinery experience has shown that in the oxydrolysis autoclaves Type 316 ELC material is slightly pitted at a rate of 0.001 to 0.005 in/yr. Pitting shows up on heating coils to a greater extent than on vessel walls. No pitting tendency had shown up at all on any austenitic stainless steels in the short pilot plant test and this indicates that a longer period than 25 days is desirable in evaluating materials for a permanent plant. If 304 had been chosen instead of 316 ELC, which would have been quite legitimate from the corrosion test, the pitting problem might be serious now.

A short test of 27 days under reduction conditions in the pilot plant also indicated that all welded and non-welded austenitic stainless steels had corrosion rates of less than 0.0005 in/yr. A test of over a year in the refinery verified these corrosion rates. However, some difficulty has been experienced with reduction autoclaves but because it is not related to corrosion tests it will be discussed later.

The erosive nature of nickel powder slurry when it is flashed from 500 psig to atmospheric pressure was not fully appreciated from pilot plant work. Some redesign of discharge piping and flash tanks was found necessary to reduce this erosion. Redesign consisted of eliminating unnecessary bends in discharge piping, changing the angle of entry into the flash tanks and experimenting with different hard surfacing materials.

Stainless steels such as Type 304 and 347 have been used in different piping modifications in the metals recovery circuit with very good success.

A pilot plant test lasting 27 days was used to evaluate materials for the sulfide precipitation of nickel and cobalt. This test showed that Type 304 and 347 stainless steels suffered weight losses up to 0.017 in/yr with pitting. Type 316 ELC showed general attack with a low rate of 0.00004 in/yr. It was chosen for most refinery equipment and has shown very little corrosion even though sulfides cake on the surface to produce crevices. Pilot conditions probably were somewhat more severe with considerable excess of hydrogen sulfide over refinery conditions, where a slight deficiency of sulfide is maintained for process reasons.

Acid leaching of cobalt-nickel sulfides with air is carried out in Type 316 ELC equipment at 250 F. pH may vary from 6½ to 7½ at the start of leaching down to 2½ to 1½ at the end. As long as an oxidizing atmosphere is maintained there is no difficulty with 316 or 316 ELC, but this can be difficult to maintain in the slurry feed line where addition of acid may create a reducing atmosphere. However, if the feed slurry is kept at a pH of 5 or above and acid added in the presence of air, no particular difficulty is encountered.

Crystallizing ammonium sulfate has been a standard process for many years. Type 316 or 316 ELC was used in the construction of Sherritt's plant as in many others and is showing no sign of corrosion.

The discussion thus far has been a general outline of refinery materials of construction as compared to pilot plant testing. Some corrosion problems will now be discussed in more detail.

Reduction Autoclaves

It was mentioned earlier that some difficulty has been experienced with reduction autoclaves. These vessels are constructed of Type 316 ELC stainless steel integrally clad on carbon steel backing material. The stainless thickness is 10 percent of the overall thickness of 1⅜ inches. One trouble not fully appreciated as a problem in the autoclave design is mechanical in nature and is caused by the difference in expansion between stainless and carbon steel. The other is a corrosion problem resulting from contamination of the stainless cladding before or during fabrication of the autoclaves.

To illustrate the mechanical problem, Figure 8 shows a stainless steel lined nozzle which feeds high pressure steam to a coil in the autoclave. Keeping in mind that the reduction process is cyclic with respect to temperature and pressure, it is easy to see what happens. When steam is turned on and later turned off, the stainless steel liner expands and then contracts more than the carbon steel backing material and sufficient stress is transmitted to weld 'A' and 'B' that after an unpredictable period of time one of these welds cracks. If it is weld 'A', solution leaks behind the liner and attacks the carbon steel. If it is weld 'B', steam at 900 lb gets behind the liner and bulges the stainless steel ring inside the autoclave and eventually cracks weld 'A' or 'C'. A good many of these nozzles have been modified to eliminate this condition.

The second problem is characterized by the appearance on the Type 316 ELC clad material of pits surrounded by an area of intergranular corrosion about one to two inches in diameter. Figure 9 shows one of these areas with several pits and intergranular areas outlined by dye penetrant. The outer circumference of these areas is evidenced by a rust-colored rim. Figure 10 is a close-up of one of these areas that has been brushed over lightly with a sander. The corrosion pattern is quite evident.

Spots such as these have shown up in about four shell courses in two autoclaves out of a total of 20 courses in the five reduction autoclaves. No spots have been noted in any of the heads. It appears, therefore, since three autoclaves are entirely free of this condition, that the corrosion is caused by some abnormal condition during the manufacturing of the material for or the fabrication of the other two autoclaves.

Trepan samples were cut out of the autoclave wall to investigate these areas metallographically. A photomicrograph showing the structure of the stainless steel from the surface to the carbon steel backing material is shown in Figure 11. Note that the cross-section of stainless is divided roughly into three equal parts with the surface area containing about 0.22 percent C, the middle section about 0.037 percent C which is normal for Type 316 ELC and the area next to the backing material about 0.074 to 0.33 percent. Carbides are precipitated in the high carbon surface area and, therefore, explain the corrosion that was taking place. However, it has not been determined just what caused the carbon contamination. Presumably it could have occurred in any one of four operations:

1. Rolling of the stainless billet;
2. Fabrication of the clad plate, which is done at about 2400 F;
3. Rolling of the clad plate into shell courses which was done at about 1600 F;
4. Stress relieving of completed vessel at 1150 F.

This surface contamination, of course, could have been prevented if proper care had been used during all these stages of fabrication.

It is interesting to note the extent to which carbon has migrated from the backing material into the stainless steel during the last three heating operations. Carbon has been increased in the stainless for a distance of about 0.045-inch from the mild steel backing material.

Copper Liquor Regenerator

It was mentioned earlier that copper ammonium formate was used to scrub the nitrogen-hydrogen mixture in the production of ammonia. It takes out most of the carbon monoxide and a good part of the carbon dioxide that remains after monoethanolamine scrubbing.

A flowsheet is shown in Figure 12 which outlines the regeneration system and the construction of the regeneration tower. Copper solution, containing carbon monoxide and carbon dioxide, is fed from the main scrubbing tower to the top of the regeneration tower where it cascades over Raschig rings and is collected on a plate half-way down the tower. It flows by gravity through two heat exchangers and a steam heater which increases its temperature from 100 to 167 F. It then flows into the top of the lower half of the tower, through more Raschig rings and into a hot storage tank. Carbon monoxide and carbon dioxide together with some ammonia are driven off as the copper solution is being heated and as it goes through the lower section of the tower. These gases, together with air which is injected in the hot storage tank, pass up the tower through the upper section where some ammonia is re-absorbed. Hot copper solution from the storage tank is pumped through the heat exchangers and is refrigerated before being used to re-absorb carbon monoxide and dioxide from the main process gas stream in the scrubbing tower.

After about two years of operation, holes developed in the shell of the tower just below the collector plate as shown in Figure 13. In fact, the tower was almost completely severed at this point with only a small amount of metal left to prevent the collapse of the top half. A band was welded around the outside of the tower to strengthen it and to seal the leaks until a new section could be fabricated and a convenient shutdown time arranged.

The exact mechanism of this corrosion has not been determined but it was theorized that condensation took place on the bottom of the collector plate because of the difference in temperature between the top and bottom sections of the tower. The condensate, washing down the wall of the vessel, caused rapid corrosion. The composition of this theoretical condensate is also very theoretical. It seems unlikely that an ammoniacal solution such as a carbonate or a formate could corrode steel that quickly but it also seems unlikely that an acid solution such as for-

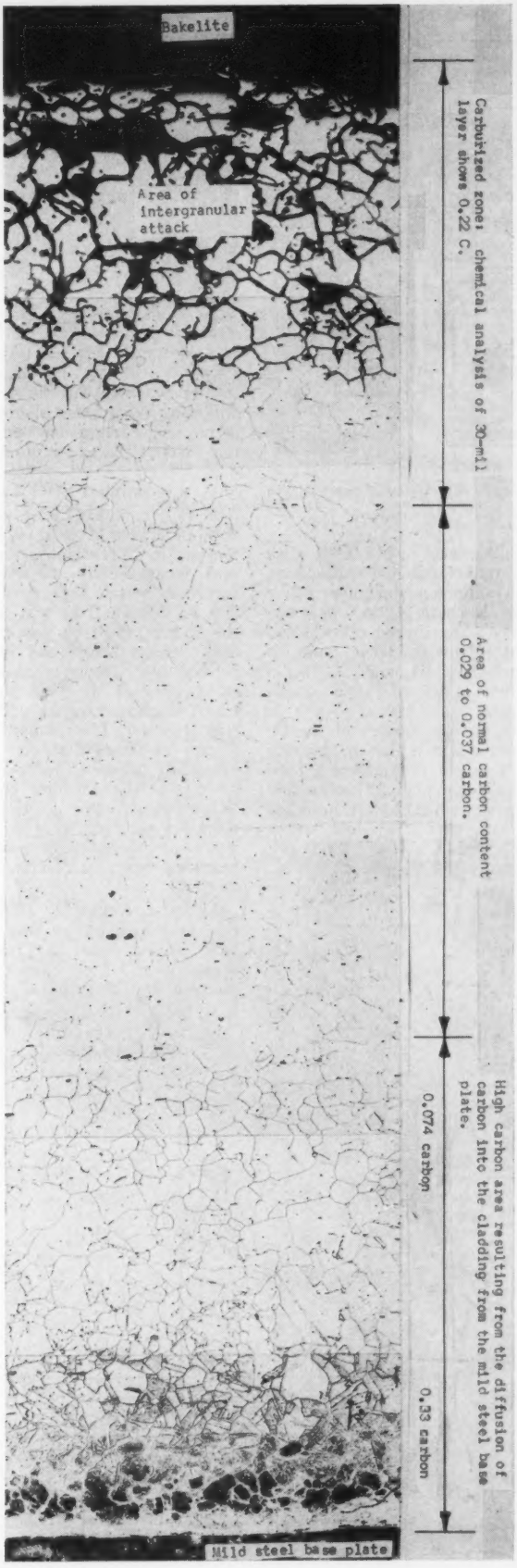
Bakelite
Area of intergranular attack
Carburized zone: chemical analysis of 30-mil layer shows 0.22 C.
Area of normal carbon content 0.029 to 0.037 carbon.
0.074 carbon
0.33 carbon
High carbon area resulting from the diffusion of carbon into the cladding from the mild steel base plate.
Mild steel base plate

Figure 11—Metallographic structure of Type 316 ELC cladding at a corrosion spot on reduction autoclave. (Courtesy Battelle Memorial Institute.) Approx. X 63. Electrolytic Sodium Cyanide etch.

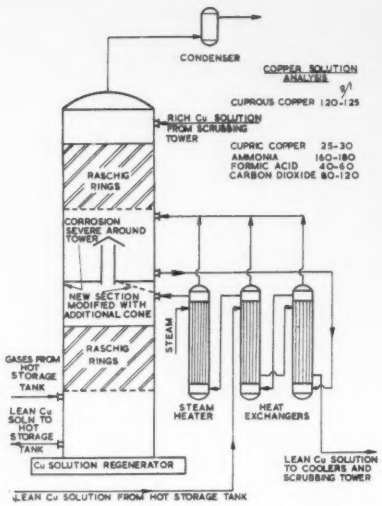

Figure 12—Schematic flow diagram of copper solution regeneration.

mic or carbonic acid could exist with considerable ammonia in the atmosphere. However, an ammonia absorption tower in the metal leaching section was corroded severely when carbon dioxide was a constituent of the gas being absorbed so it is known that NH_3 and CO_2 are a potent mixture under the right conditions.

When the corroded section of the regeneration tower was replaced, an additional plate was put in to form a cone below the collector plate. This created an insulating space which was supposed to prevent any further condensation. After two more years of operation, there is no severe corrosion taking place on the walls of the new section.

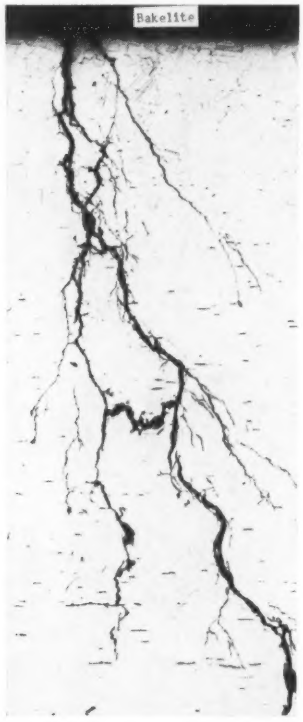

Figure 14—Stress-corrosion cracking in a Type 316 ELC stainless steel tank. 50 X. Modified aqua regia etch.

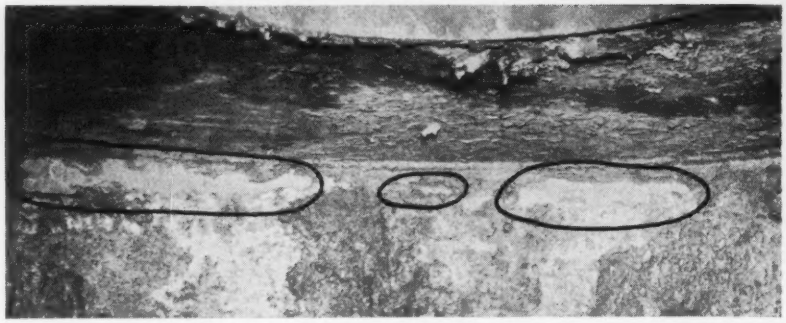

Figure 13—Corrosion and scaling in copper solution regenerator.

Stainless Steel Tank

A rather unusual case of stress corrosion cracking occured on the bottom of a type 316 ELC stainless steel tank. This tank, located inside the reduction building on a concrete base, operated at about 200 F. When a leak was discovered on the bottom, the tank was raised and the base cleaned before any deposits could be examined. However, about a third of the bottom of the tank showed cracks which originated on the underside of the tank bottom. The photomicrograph in Figure 14 shows that the cracking was caused by stress-corrosion. Since deposits on top of the concrete base were not examined, it is not possible to say that chlorides were or were not present. If they were there, they could have come from any one or a combination of three sources.

1. Water, containing a few ppm chloride and used in washing floors could have leaked under the tank on many occasions and been evaporated to concentrate the chlorides.

2. A less likely possibility is that process solution could have done the same.

3. Attack was evident on the concrete under the tank. This could have been caused after the tank started leaking but, if any of the concrete was leached by water, some chloride could have been contributed from this source. If sufficient time becomes available, more work will be done to try to determine the exact cause of this failure.

Two rather common corrosion problems are probably worth mentioning briefly. The first of these is underground piping corrosion. A few leaks have occurred on water piping and one on gas piping in the last two years. Examination of these pipes and others exposed during plant expansion has shown that, in certain areas, pitting is a problem. A partial cathodic protection system has been installed to alleviate the corrosion condition.

The second common corrosion problem is associated with recirculating cooling water. Until last year there was always a difficulty in maintaining the desired pH for phosphate treatment because of ammonia pickup into the system. At times it was not practical to add the necessary sulfuric acid to keep

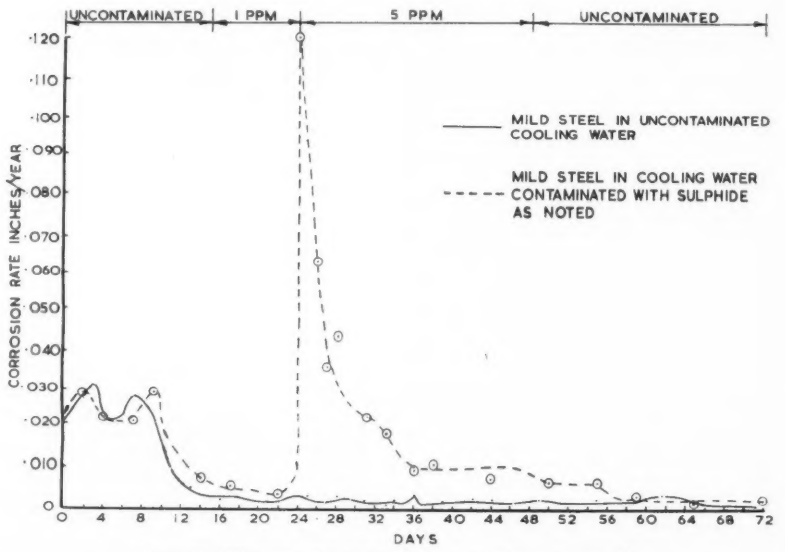

Figure 15—Effect of sulfide contamination on cooling water corrosion.

the pH down so control was attempted at a pH as high as eight. Occasionally, contamination by other known materials such as monoethanolamine and hydrogen sulfide has contributed to accelerated corrosion. An electrical resistance corrosion measuring device is being used to follow cooling water corrosion at several points in the system so that immediate attention can be given to any water stream showing a high corrosion rate. Tests are also being carried out to determine the effect of various contaminants on the water corrosion rate. Figure 15 shows the effect of sulfide contamination on the corrosion rate of mild steel in cooling water. The water is exactly the same in both cases except for sulfide contamination.

Similar experiments are planned for other contaminants such as ammonia, ammonium sulfate, monoethanolamine, etc. Effectiveness of other water treatment methods also may be investigated before the program is completed.

Other experiments on stress corrosion and contamination of stainless steel have been carried out and work is progressing on the evaluation of different plastics and paints under various plant conditions.

Acknowledgment

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Any discussion of this article not published above will appear in the December, 1959 issue.

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Simple Screening Test for

Determination of Inhibitor Film Persistence*

By JOHN A. KNOX and ROY STOUT

Abstract

Describes a simple screening test similar to copper ion displacement test which has been devised to screen corrosion inhibitors to be used in the inhibitor squeeze process. This copper ion test evaluates durability of protective inhibitor films formed by relatively high inhibitor concentrations rather than the usual test's determinations at low concentrations. Discusses effect of inhibitor concentrations, adsorption time, adsorption temperature, adsorption from various solvents and coupon surface preparation on film persistence of chemical compounds considered to have inhibiting properties. Evaluation is based on time required for copper to form on coupon. 5.8.4

Knox

Stout

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Introduction

INHIBITOR SQUEEZE as a method of applying corrosion inhibitors to oil well equipment has become so widely used that a simple testing procedure for screening inhibitors was needed. Most of the conventional corrosion tests are designed to determine the relative effectiveness of inhibitors at low concentrations.

Value of the inhibitor squeeze process depends on the formation of a durable protective film on well equipment as a high concentration of inhibitor is pumped into the well. The extended copper ion displacement test is a method for determining durability of the film formed on metal by a relatively concentrated inhibitor solution.

A similar test called the copper ion displacement test applies to low concentrations of inhibitors.[1]

For the purpose of this test the copper sulfate solution was considered only as a corrosive medium. The pH of the solution was 3.18. The reaction, $Fe + Cu^{++} \rightarrow Fe^{++} + Cu$, has a standard electrode potential of 0.7848 volts which acts as a driving force to cause the copper to be displaced from solution by the iron.

A copper sulfate solution is a very corrosive environment for iron. To prevent the deposition of copper, a stable impermeable film must be present on the iron surface. A film of this type does form with certain inhibitors, and long term protection from the deposition of copper is shown by them. Once the film is penetrated by the copper, plating proceeds rapidly, resulting in a complete breakdown of the film. Under proper conditions, inhibitor films have been observed which do not break down within the time limits of this test.

Photomicrographic movies of copper plating on an inhibited and uninhibited metal surface have been made.

Formation of metal complexes between amines, etc., and copper may be a source of error in this test. This is not completely ruled out, but most of the transition elements and several others including iron and copper form the Werner type coordination complexes with amines.[2] Hence, iron can complex with the amines in a similar manner to copper. This complexing may in some way explain the effectiveness of amine compounds as inhibitors for iron. Actually, the metal-amine complexes should form best when both the metal compound and amine are in solution. The compounds used in these tests are not mutually soluble to any great extent in the solvents used; therefore, the reaction should proceed only with great difficulty.[3]

If the complex compounds in each instance do form and adsorb on the metal surface, their size and shape may cause one inhibitor to work well and cover the surface completely while another leaves a permeable film through which copper can migrate to the metal surface.

Experimental Work

Materials

Equipment required for each test run in the experiment included three 50-milliliter beakers and one glass "S" hook.

Test coupons used were made of 1010 mild, cold-rolled, steel strip, 1/16-inch by ½ inch. The steel strip was sheared into 1 inch lengths, and 3/16 inch holes were punched in the end of each coupon.

For test coupon surface preparation, all coupons were washed in acetone, stored under acetone and air dried before using. The standard test coupons for Test A conditions were tumbled 24 hours in a rotating vessel with 10-30 mesh Ottawa sand. The following three alternate methods of preparation also were used:

Untreated: Coupons were used as received. There was no additional surface preparation. No rust or mill scale was observed on the coupons.

Acidized: Coupons were immersed in 15 percent uninhibited hydrochloric acid for 5 minutes and washed with tap water.

Sand Tumbled and Polished: Sand tumbled coupons were polished with a high speed wire wheel.

Eight Reagents were used as discussed below:

Salt Solution: 5 percent technical grade soduim chloride by weight in distilled water.

Copper Sulfate Solution: 10 percent technical grade $CuSO_4 \cdot 5H_2O$ by weight in distilled water.

Calcium Chloride Solution: 5 percent USP grade $CaCl_2 \cdot 2H_2O$ by weight in distilled water.

Purified Kerosene: No special cut of kerosene was taken, but it was purified by treatment with a mixture of Fuller's Earth and decolorizing charcoal.

Petroleum Ether: 30 to 60 C boiling point meets ACS specifications.

Xylene: Technical grade. Boiling point —138 to 140 C.

Mineral Oil: Heavy white USP mineral oil with viscosity approximately 70 cps at 100 F.

Compounds Tested: The compounds tested were chosen because they were thought to have inhibiting properties. Many of them have been studied, and data on their properties can be found in the literature. They vary in functional groups, saturation and chemical nature, but for the most part have at least one 18-carbon chain per molecule.

All are available commercially with the exception of the salts. The materials were used as received from their manufacturer. Salts were prepared by mixing the amine with the acid on the basis of their combining molecular weights indicated by the manufacturer. Temperature during the salt preparation was not allowed to rise above 140 F. The chemicals tested are listed in Table 1.

At all concentrations tested, Compounds 1, 2, 3 and 17 were partially insoluble in all solvents.

At 25 percent concentration in kerosene, Compounds 5, 10, 11, 12, 13 and 14 were not completely soluble. Compound 8 increased the viscosity of the kerosene.

Procedures

In the extended copper ion displacement test, a test coupon attached to a glass hook was immersed in 5 percent

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TABLE 1—Results of Extended Copper Ion Displacement Test

% Active Ingredient	Property Varied / Variation	Test A Basic Test	Test B Inhib. Cone. % 0.01	0.1	25	Test C Cone. 0.1% Time 1 min	30 min	Test D Time 1 min	30 min	Test E Temperature 100°F	150°F	Test F Solvent Pet. Ether	Xylene	Mineral oil	Test G Coupon Pretreatment Un-treated	Acid-ized	Polish	Test H Brine 5% $CaCl_2$	NACE Test* % Protection
97	1. Hydrogenated Tallow Amine	-1/8	-1/8	-1/8	2	-1/8	-1/2	-1/8	-1/2	-1/8	-1/8	-1/8	-1/8	-1/8	-1/8	-1/8	-1/8	-1/8	98
87	2. Tallow (Primary) Amine	-1/8	-1/8	-1/8	-1/8	-1/8	1/2	-1/8	1/2	-1/8	-1/8	-1/8	1/8	-1/8	4	-1/8	-1/8	-1/8	98
80	3. N-Tallow Propylene diamine	-1/8	-1/8	-1/8	360	-1/8	-1/8	-1/8	-1/8	-1/8	-1/8	-1/8	1/8	-1/8	-1/8	-1/8	-1/8	-1/8	92
50	4. Tallow Trimethyl Ammonium Chloride	-1/8	-1/8	-1/8	-1/8	-1/8	-1/8	-1/8	-1/8	-1/8	-1/8	-1/8	-1/8	-1/8	-1/8	-1/8	-1/8	-1/8	91
60	5. 1 (2 Hydroxyethyl) 2 Heptadecenyl 2 Imidazoline	-1/8	-1/8	-1/8	-1/8	-1/8	-1/8	-1/8	-1/8	-1/8	-1/8	-1/8	-1/8	-1/8	1/2	-1/8	-1/8	-1/8	100
100	6. Polyoxyethylene Tallow Amine (5 Moles Ethylene Oxide)	-1/8	-1/8	-1/8	-1/8	-1/8	-1/8	-1/8	-1/8	-1/8	-1/8	-1/8	-1/8	-1/8	-1/8	-1/8	-1/8	-1/8	98
100	7. USP Oleic Acid	-1/8	-1/8	-1/8	-1/8	-1/8	-1/8	-1/8	-1/8	-1/8	-1/8	-1/8	-1/8	-1/8	1/8	1/8	-1/8	-1/8	0
62	8. Sodium Salt of Long Carbon Chain Sulfonic Acid	-1/8	-1/8	-1/8	-1/8	-1/8	-1/8	-1/8	-1/8	-1/8	-1/8	-1/8	-1/8	1/8	-1/8	-1/8	-1/8	-1/8	4
100	9. Polyoxyethylene Sorbitol Hexaoleate (30 moles Ethylene Oxide)	-1/8	-1/8	-1/8	-1/8	-1/8	-1/8	-1/8	-1/2	-1/8	-1/8	-1/8	-1/8	-1/8	-1/8	-1/8	-1/8	-1/8	51
80	10. N Tallow 3 Amino Propylene Ammonium Oleate	3	-1/8	-1/8	360	-1/8	2	3/4	3	6	28	-1/8	3	2	1/2	1/2	1	3	79
80	11. N Tallow 3 Amino Propylene Ammonium Dioleate	3	-1/8	-1/8	360	-1/8	2	2	12	36	69	-1/8	3	7	1/8	3/8	2	3	82
87	12. Tallow Ammonium Oleate	4	-1/8	-1/8	+624	-1/8	4	3	4	5	-22	-1/8	13	15	2	2	2	3/8	93
87	13. Tallow Ammonium Dioleate	7	-1/8	-1/8	+624	-1/8	4	2	8	25	-22	-1/8	6	12	3	2	2	3/8	96
62	14. 1(2Hydroxyethyl)2 Heptadecenyl;2 - Imidazoline - Oleate	3/4	-1/8	-1/8	-96	-1/8	4	1/4	1	-22	-22	-1/8	.2	15	-1/8	-1/8	-1/8	3/4	97
100	15. Polyoxyethylene Tallow Ammonium Oleate (5 Moles Ethylene Oxide	3/4	-1/8	-1/8	-96	-1/8	4	1/2	5	+528	336	-1/8	1/8	-1/8	-1/8	-1/8	3/8	3/4	98
50	16. Inhibitor A	55	-1/8	5	+360	-1/8	15	35	55	144	88	-1/8	3	22	25	35	56	55	99
50	17. Inhibitor B	42	-1/8	-1/8	66	-1/8	5	20	46	79	95	-1/8	3	14	24	24	24	5	96
	18. Blank	-1/8	-1/8	-1/8	-1/8	-1/8	-1/8	-1/8	-1/8	-1/8	-1/8	-1/8	-1/8	-1/8	-1/8	-1/8	-1/8	-1/8	

Note: Time given in hours till copper plated coupon.
Tests run under following conditions (Conditions for Test A) unless otherwise specified:
1. Soaked in 5 percent aqueous salt solution 5 minutes.
2. Soaked in 1 percent inhibitor solution in purified kerosene 5 minutes.
3. Soaked in 10 percent aqueous copper sulfate solution until copper plates.
4. Temperature for tests was 77 F.

* Run according to standard static NACE(®) test using H_2S brine—Inhibitor Concentration = 100 ppm. Soaking Time of Coupon = 30 seconds.

aqueous sodium chloride for 5 minutes. Following the brine exposure, the coupon was immersed in a 1 percent solution of the inhibitor in purified kerosene for 5 minutes. The coupon was transferred to a 10 percent aqueous solution of copper sulfate where it remained until copper plating was noted. Tests were run at 77 F unless otherwise specified. Surface of the test coupon was prepared by tumbling with sand for 24 hours. This is a description of the basic test or Test A. Variations in the test will be described later.

All compounds have been tested using the standard NACE corrosion test for screening inhibitors.[4] Inhibitor concentration used was 100 parts per million. Test coupons used were the sand tumbled type described previously. They were soaked in the oil containing inhibitor for 30 seconds.

Potential curves of copper plating on the test coupons were recorded of the copper-iron system in 10 percent copper sulfate solution. A copper anode and iron cathode were used. The cell potential was fed to a recorder through a high resistance shunt.

Photomicrographic movies of inhibitor film breakdown and copper deposition were taken at X100 magnification with a 16mm movie camera at 8 frames per second. A small glass cell made with microscope slides held the metal sample and treating solution while the photomicrographs were taken. The sample was kept off the bottom of the cell by a short section of thin glass under each end so the fluid could surround the coupon.

Experimental Results

Results of the extended copper ion displacement tests are shown in Table 1. The table is divided into Tests A through H. Test A (described above) is the basic test, and all others are compared to it. Only one parameter of Test A is varied in each of the other tests, with the exception of Test C in which two properties were varied. The properties varied in each test were inhibitor concentration, time, temperature, solvent and coupon pre-treatment as shown in Table 1.

No attempt was made to determine quantitatively the amount of copper deposited on the coupons. Breakdown time or time required for copper to plate varied enough between inhibitors that the time was considered as the measure of the effectiveness.

The 17 compounds tested on the copper ion test also have been tested using the standard procedure of the NACE for screening inhibitors. Results are reported as percent protection in the last column in Table 1.

Figure 1 is a typical potential-time curve obtained when a coupon was treated with inhibitor solution for various lengths of time then exposed to a copper sulfate solution. Length of the plateau of the curve is a measure of the length of time that the inhibitor film is effective. Copper deposition on the coupon could be seen soon after the break in the plateau.

Cinemicrographs were taken of the metal samples under kerosene containing no inhibitor and as copper sulfate solution was added. The immediate plating action and rapid deposition of copper could be seen. A metal sample treated with inhibitor also was photographed and the durability of the inhibitor film shown. No copper deposit was observed for 4 hours and 45 minutes.

After the first spot of copper appeared, the test coupon was covered within 15 to 20 minutes. A black deposit was noted building up in the pits on the test coupon over the 4 hour and 45 minute period. No attempt was made to explain this phenomena; however it is of interest to note that the copper did not deposit first at these points.

Discussion

Compounds 1 through 9 showed no tendency to form protective films on the surface of the test coupons under any of the test conditions. One exception was Compound 3 at 25 percent concentration. These materials were the amines, quaternaries, imidazolines, fatty acids, ethylene oxide derivatives and sodium sulfonic acids.

Compounds 10 through 17 exhibited varying degrees of inhibitor film persistence. For the most part they included salts of amines, etc., and oleic acid.

Each test condition varied caused a variation in the results.

Concentration of the inhibitor was very important. Some optimum concentration must be chosen for the test to differentiate between the inhibitors.

Time of adsorption did not affect the results a great deal if it was five minutes or more. At one minute, soaking time optimum results were not obtained.

Temperature of adsorption also was important. Only a few degrees temperature difference caused a marked difference in the time copper started to plate.

The solvent used to carry the inhibitor caused considerable variation in film breakdown time. Petroleum ether may be a good solvent for copper or iron com-

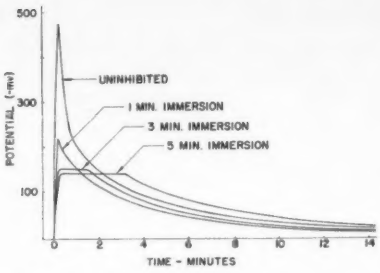

Figure 1—Typical potential-time curves obtained with a 0.1 percent inhibitor solution.

plexes.[4] The viscosity also may be a factor. Because of solvent action, adsorption from xylene may be less than from kerosene. Mobility of the inhibitor molecules in mineral oil may be lower than in the other solvents and complete oil wetting of the coupon may not be obtained after being exposed to brine.

The three types of surface preparation used in Test G showed little variation in breakdown time for a particular inhibitor. The sand tumbled coupons used in Test A generally gave longer breakdown time.

Difficulty in obtaining uniform surface preparation may account for variations found throughout the testing program. Formation of thin oxide films[5] on the samples, surface deformities and the presence of tiny sand particles observed in the coupon surfaces may contribute to inconsistency.

Substitution of calcium chloride brine for sodium chloride brine indicated the calcium chloride brine affected the persistence of the inhibitor film.

Duplicate results for any set of test conditions were difficult to obtain. The trend of inhibitor efficiency remained the same although the actual time for copper to plate varied.

Correlation between the standard NACE test and the extended copper ion test is not evident from the results.

Only preliminary data has been obtained by the potentiometric method, but the results indicate this may be a method of obtaining data on film persistency. The circuitry is simple, and the same corrosive media is used throughout the tests. Correlation is obtain between adsorption time and film life.[6]

Summary

A simple qualitative screening test has been presented for the determination of inhibitor film persistence.

Effectiveness of an inhibitor in commercial use may vary from well to well and field to field. This test shows that practically any condition changed will have a marked effect on the results.

The work has not been completed. Several conditions of the test must be evaluated further. Temperature, concentration and surface preparation all require more study.

The test is submitted for consideration in an effort to get more laboratories interested in the test so that its full potential can be utilized.

Acknowledgment

We would like to acknowledge the assistance of David Morris and Bill Billings for conducting the bulk of the laboratory research.

Warren Philipp, now with Lewis Flight Laboratories in Cleveland, Ohio, also contributed to the article.

An expression of thanks goes to Halliburton Oil Well Cementing Company for permission to publish this article.

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6. E. J. Simmons. Use of the Pearson Bridge in Corrosion Inhibitor Evaluation. *Corrosion*, 11, 255t (1955) June.

DISCUSSIONS

Comments by E. R. Allen, Jr., Humble Pipe Line Company, Houston, Texas:

Method described measures persistence in aqueous solution. Can method be adapted to measure persistence in oil solutions?

Reply by John A. Knox:

The coupon could probably be washed several times in oil after being treated with inhibitor solution and before placing in the copper sulfate solution. This may give some indication of the ability of an inhibitor to stay on a metal surface in the presence of oil.

Comment by Parke Denton Muir, Tulsa, Oklahoma:

When the temperature was varied, were all of the solutions kept at the elevated temperatures or was only the inhibitor-kerosene solution kept at the elevated temperature?

Reply by John A. Knox:

The solution of inhibitor in kerosene was the only solution which was heated when the temperature was varied. The salt and copper sulfate solutions were kept at approximately 77 F.

Any discussion of this article not published above will appear in the December, 1959 issue.

Other Technical Information Published in Corrosion

Each issue of CORROSION includes four or five technical articles of immediate, practical interest in the "Technical Topics" section. The articles in this section and authors will be included in the 1959 Corrosion Index which will be published in the December issue.

Project of NACE Committee T-2

Minimum Requirements for Protection Of Buried Steel Pipe Lines

By W. H. Stewart
Sun Pipe Line Co., Beaumont, Texas
T-2 Chairman

Concurrent with the re-organization of the NACE Technical Committee set-up and formation of Group Committee T-2 in 1954, the task of formulating basic requirements for underground pipe protection by NACE was requested by Subcommittee 8 of ASA B31.

As a result of this correspondence between NACE and ASA and of discussions within NACE Technical Practices Committee, the chore was delegated to Group Committee T-2 with instructions to form a task group from its ranks for this undertaking.

In these discussions, where policy and ultimate goals to be attained were aired, it was repeatedly emphasized that **"minimum** requirements" would be the result and **not** "specifications" or "standards." This policy did not stem from any hasty or ill-chosen desire to avoid the issue. The ASA had previously formulated codes on various physical properties and construction practices on underground gas piping. This, compared to corrosion prevention methods, was a relatively simple task.

Conversely, in the pipe protection field no such standards on material and application had been established as had been in pipe lines and their fabrication by API, AGA and other organizations.

It was concluded that NACE should establish "minimum requirements" in this field and form a base from which further design or improvements would doubtlessly be instituted by those concerned with the individual job.

Many reasons exist in support of such a decision. Geographically, the requirements vary from almost none in a few isolated areas to extreme severity in others. Numerous methods of coating with various types of materials have been used with some degree of efficacy in different sections of the country. It becomes obvious, therefore, that no brief, concise "standard specification" that would severely limit the type of material and application could be tolerated or accepted. Because the science of corrosion prevention is not a static art, it would be undesirable to place an inflexible control over its use.

Our efforts, therefore, had to be limited to an extremely broad coverage of the subject with an ultimate production that everyone could live with. Naturally, somewhere along the line some product or method of limited use might be questioned as to its inclusion. The Technical Practices Committee ruled on this point in one decision, finding that a unit committee could accept or reject such a material proposed for inclusion in its recommendations.

Some misunderstanding exists outside Committee T-2 concerning "specifications." It should be thoroughly understood that this report or recommendation (Minimum Requirements) is neither a "standard" nor a "specification." True, some of the completed recommendations contain certain base tables or material specifications, but these are considered "minima" and are compiled in such a manner as to include several existing materials of varying divergence in formulation, all to some degree superior to such specification. Any reference to these complete reports, printed or otherwise, as being NACE specifications is, to put it mildly, misleading.

To date, "minimum requirements" have been completed on asphalt coatings and coal tar coatings. Shields and wrappers is near completion, and minimum requirements for cathodic protection is likewise being finalized.

Once the complete picture, as briefly outlined here, is understood, confused thinking and outright opposition from a few sources should disappear. The need for some basic level in this field exists and must be met. The proper source is one that has the "know how" and can formulate such a base that can be lived with by the industry. Rest assured that other organizations, lacking such a guide, may easily turn to some source that could create codes extremely difficult, even nigh impossible to meet.

Canadian Region News

Edmonton Section heard E. H. Hochhausen of Rice Engineering & Operating, Ltd., speak on plastic piping in the oilfields at the August 27 meeting.

The October 22 section meeting will be held in the Corona Hotel.

•

Montreal Section at a recent meeting discussed the corrosion problem caused when street cars were removed from Montreal streets. Cathodic protection systems had been connected to the street car tracks. Another system of grounding must be found.

•

Canadian Region Eastern Division will hold its 1960 Conference January 17-20 in Toronto. H. A. Webster of Corrosion Service Ltd., Toronto, will be conference chairman.

Other committee chairmen for the conference will be Frank Farrer, registration, Trans-Northern Pipe Line Co., Toronto; D. J. Cowan, finance committee, Corrosion Service Ltd., Toronto; and Gus Gillrie, publicity, Oil in Canada, Toronto.

•

The 1959 North Central Region Conference will be held October 20-22 in Cleveland, Ohio.

All Booths Are Sold For 1960 Corrosion Show to Be in Dallas

All booths have been sold for the 1960 Corrosion Show, six months before the show is to be held March 15-17 in conjunction with the 16th Annual NACE Conference in Dallas, Texas.

This is the earliest date before a corrosion show that all available booth space has been sold, according to R. W. Huff, Jr., NACE Exhibits Manager.

Eighty-eight companies will have diversified displays of corrosion control materials, equipment and processes in the 121 booths at the Dallas Memorial Auditorium. Booths have been arranged to give best traffic flow to as many booths as possible. Technical meetings of the conference will be held on the two floors above the exhibit area.

A list of exhibitors is given below:

Agra Engineering Company
Aluminum Company of America
Amercoat Corporation
American Smelting & Refining Company
Armco Steel Corporation
Black, Sivalls & Bryson, Inc.
Brance-Krachy Co., Inc.
Stewart R. Browne Mfg. Co., Inc.
Burndy Corporation
A. M. Byers Co., Inc.
Carboline Company
Cameron Iron Works, Inc.
Philip Carey Mfg. Co.
Carpenter Steel Co.
Cathodic Protection Service
Central Plastics Co.
Centriline Corporation
Chicago Bridge & Iron Co.
Clementina, Ltd.
Clemtex, Ltd.
Corrosion Rectifying Co.
Coast Paint & Lacquer Co.
Cosasco Division—Grant Oil Tool
Crane Company
M. J. Crose Mfg. Co., Inc.
Crucible Steel Co. of America
DeVilbiss Company
Dia-Log Company
John L. Doré Co.
Dow Chemical Company
Dresser Mfg. Div.
Duriron Co., Inc.
Fansteel Metallurgical Corp.
Fibercast Co.
R. C. Foltz Company
Garlock Packing Co.
General Mills, Inc.
Glidden Company
Good-All Electric Mfg. Co.
Gulf States Asphalt Company, Inc.
Harco Corporation
Heil Process Equipment Corp.
Hercules Powder Company
International Nickel Co., Inc.
Johns-Manville Sales Corporation
Koppers Company, Inc.
Kraloy Plastic Pipe Co., Inc.
Lincoln Engineering Company
F. H. Maloney Company
Metal-Cladding, Inc.
Nicolet Industries, Inc.
Orchard Paper Co.
Otis Engineering Corp.
Owens-Corning Fiberglas Corp.
Permacel
Pfaudler Co.
Chas. Pfizer & Co., Inc.
Phelps Dodge Copper Products Corp.
Pipe Linings
Pittsburgh Coke & Chemical Company
Pittsburgh Corning Corporation
Plicoflex, Inc.
Minnesota Mining & Mfg. Co.
National Carbon Company
Kendall Co., Polyken Sales Division
Radio Receptor Co., Inc.
Reilly Tar & Chemical Corporation
Republic Steel Corp.
Resistoflex Corp.
Rio Engineering Co.
Rockcote Paint Company
Royston Laboratories, Inc.
Sentinel Chemicals, Inc.
Shell Chemical Corporation
Shell Oil Company
Socony Paint Products Company
Standard Magnesium Corporation
D. E. Stearns Company
The Tapecoat Co.
Tejas Plastics Materials Supply Company
Texsteam Corporation
Tinker & Rasor
Truscon Laboratories
Tubular Lining Corporation
U. S. Stoneware Company
Victaulic Company of America
Visco Products Company
T. D. Williamson, Inc.

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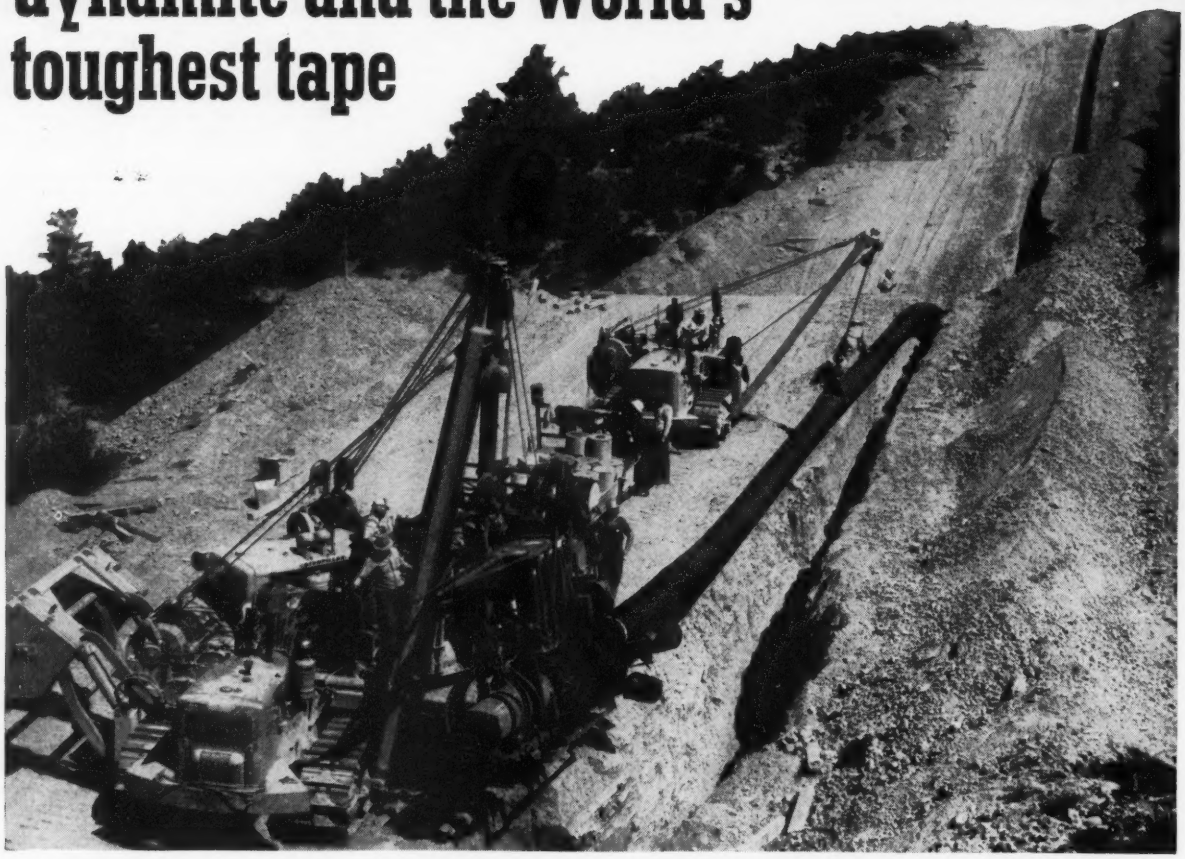

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Plainfield, New Jersey
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San Francisco, Calif.
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San Francisco, Calif.
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Farwest Corrosion Control Corp.

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South Central Region News

Denver Program Completed For Oct. 12-15 Conference

Final plans are complete for the October 12-15 South Central Region Conference in Denver, Colo. Registration fee for members will be $15; $17 for non-members; $7.50 for lady guests accompanying a conference registrant. No additional charge will be made for any function listed on the official program.

Conference activities will be held in the Cosmopolitan and Brown Palace Hotels.

List of the technical papers to be presented, abstracts of the papers and biographical sketches and photographs of the authors were published on pages 80-82 of the September issue of CORROSION.

Ladies Program

The Cosmopolitan Hotel's Club Room has been reserved as the ladies social room during the conference.

A bus trip to Colorado Springs, the Air Force Academy, Manitou Springs and luncheon at the Broadmoor Hotel is planned for Tuesday, October 13. On Wednesday, October 14, a fashion show and luncheon will be held in the Cosmopolitan Hotel Century Room. Individual tours to the Denver mint, Museum of Natural History and other points of interest in Denver will be scheduled.

Dinner and Dance

Special dinner show and dance are scheduled for Wednesday evening, October 15, in the Cosmopolitan Hotel Silver Glade Room.

At the annual business luncheon, Wednesday, October 14, Chester Lauch, the "Lum" of Lum and Abner fame, will appear.

•

Nineteen technical committee meetings are scheduled for the South Central Region Conference, October 12-15, in Denver, Colorado.

Alamo Section held its annual social function September 15. Members' wives were invited to an informal dinner. No business was transacted.

•

Houston Section heard M. J. Olive, chief corrosion engineer with Arkansas Fuel Oil Corp., Shreveport, La., speak on corrosion and its prevention in service station underground equipment at the September 8 meeting.

No section meeting will be held this month because of the October 12-15 South Central Region Conference in Denver.

A joint meeting of the Houston Section and the Southwest District of ASTM is planned for February 9, in honor of Frank L. LaQue, ASTM president and also former NACE president.

•

Houston Section has scheduled its 5th Annual Corrosion Control Short Course for January 22 and 23 to be held at the University of Houston.

The course will cover fundamentals of corrosion, cathodic protection, maintenance and operation of corrosion preventive systems including rectifiers and galvanic anodes.

Chairman of the course is M. A. Riordan, Rio Engineering Co., P. O. Box 6036, Houston 6, Texas.

Western Region News

San Francisco Bay Area Section will hold a joint meeting October 5 with the American Society for Metals at Spenger's Fish Grotto in Berkeley. Frank L. LaQue, International Nickel Co., Inc., New York City, will be guest speaker.

William W. Kurz, president of Vacu-Blast Company, Inc., Belmont, Cal., spoke on modern blast cleaning for corrosion control at the September 8 meeting.

Northeast Region News

Pittsburg Section Sets '59-60 Dates and Program

Program dates and guest speakers for the 1959-60 meetings have been set by the Pittsburgh Section.

A coatings Roundtable Discussion with five speakers is planned for the November 5 meeting. Speakers are Kenneth Tator (roundtable chairman), Kenneth Tator Associates, J. H. Cogshall, Pennsalt Chemical Co., W. F. Fair, Jr., Koppers Co., John Keane, Steel Structures Painting Council, and G. W. Seagran, Stoner-Mudge Co.

At the October 1 section meeting, Harry Gilbert of Pittsburgh Plate Glass Company spoke on general maintenance point coatings.

Program schedule for the year follows:

December 3: Underground Roundtable Discussion with Chairman F. E. Costanzo, Manufacturers Light & Heat, T. S. Watson, Socony Mobil Oil Co., C. M. Rutter, Equitable Gas Co., D. A. Tefankjian, Texas Eastern Transmission Co., C. W. Beggs, Public Service Electric & Gas Co., and E. K. Benson, Bell Telephone Co.

January 7: Micro-biological Studies of Pipeline Back-Filled Ditches, by J. O. Harris, Kansas State University.

February 4: Modern Architectural Design and Associated Corrosion Problems: by R. A. Fisher, Carnegie Institute of Technology.

March 3: Design for Corrosion Prevention in the Process Industry, by F. L. Whitney, Jr., Monsanto Chemical Co.

April 7: Ladies Night.

May 5: Plant tour of U. S. Steel's National Tube Division, McKeesport, Pa.

Procedure in Selection of Persons to Receive Whitney and Speller Awards

Procedure in the Selection of Persons to Receive the Willis Rodney Whitney and Frank Newman Speller Awards as Approved by the Board of Directors, December 4, 1953 and Revised March 6, 1955 and December 7, 1957.

The Board of Directors of the National Association of Corrosion Engineers in a regular meeting on January 17, 1947 authorized the presentation annually of two awards by the Association:

The Willis Rodney Whitney Award is given in recognition of public contributions to the science of corrosion. A contribution to science is defined as the development of a more satisfactory theory which contributes to a more fundamental understanding of corrosion phenomena.

The Frank Newman Speller Award is given in recognition of public contributions to corrosion engineering. A contribution to enginering is defined as the development or improvement of a method, apparatus or material by which the control of corrosion is facilitated or made less costly.

Not later than January 1st of each year, the President in consultation with the immediate past President shall appoint an awards committee. This committee shall consist of at least six members with the President serving as Chairman. He shall continue to serve as chairman of the committee for the calendar year and shall present the award to the recipients selected by his committee. In the event the President upon becoming past President should be unable to serve, the President (for the year the award is to be made) shall appoint a Chairman. The other members of the committee should be outstanding members of the Association. Members shall be appointed for a three-year term with two members being replaced each year. Any member who is lost through resignation or for any other reason, shall be replaced by appointment by the President of the Association.

The Chairman of the Award Committee shall arrange to keep reasonably detailed records of the activities and discussions of the Committee. These records shall be made available to succeeding Awards Committees.

The Awards Committee shall meet at the Annual Conference, or in the event the Annual Conference is not held or is held later than June 1st the Chairman shall conduct the commitee business by correspondence.

The list of candidates to be considered for the Awards shall include:

A. The names of those proposed by the Chairmen of the Regional Divisions in accordance with the wishes of the members of the Division. Nominations may be made by local sections or may be proposed directly to the Chairman of a Region by individual members. In any case, nominations should be accompanied by an appropriate statement of the basis of the proposal. This statement shall be reproduced and submitted to all members of the Awards Committee. The Committee should have such statement for all nominees to be considered.

Prior to February 1st of each year the Regional Chairman shall forward to the Executive-Secretary all names proposed for awards, accompanied in each case by summaries of the reasons presented in support of each proposal but with no indication of the number of duplicate nominations of any individual. The object of this latter provision is to insure that awards will be made on a basis of accomplishments and other qualifications rather than by any semblance of a popular vote.

Not later than October 1st (or the publication date of the October issue of CORROSION) of each year the Executive Secretary shall notify the membership through publication in CORROSION of their privileges in this regard as set forth here. A similar notice shall be mailed by direct letter to all region and section chairmen by October 1st.

B. Any names proposed by the members of the Committee prior to February 1st of each year.

C. The Committee may review and consider nominees from previous years.

D. All nominations shall be submitted to the Executive Secretary who shall distribute copies to all members of the Committee. Nominations will be closed on February 1st.

At the discretion of the Committee members the resultant list of candidates may be reduced to a workable number by elimination of those known to lack the necessary qualifications.

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As a basis for further consideration by the Committee, the headquarters staff of the Association shall provide the Committee, through its Chairman, with a record of the contributions of each final candidate in the form of a list of his published papers and other works. Such records available in the headquarters office may be supplemented by additional information on the candidates' qualifications which may be secured by any member of the Committee from other sources, such as friends of the candidates, and especially from the proposers of the candidates who should be asked to provide their reasons for suggesting them and a summary of their accomplishments. Ordinarily, the candidates should not be made aware that they are being considered for awards by seeking such information from them directly.

The required activity of the headquarters staff should be facilitated by appropriate use of the literature abstract filing system.

The list of candidates about whom the headquarters staff is to provide the information described should be sent by the Chairman of the Committee to the Executive Secretary of the Association and to each member of the Committee not later than May 1st.

The information to be provided by the headquarters staff should be sent by the Executive Secretary to the Chairman and each member of the Committee not later than July 1st.

Information secured by any member of the Committee concerning any candidate or candidates should be sent to the Executive Secretary not later than June 15th so that it can be circulated amongst all the members of the Committee not later than July 1st.

Not later than August 1st, each member of the Committee shall submit to the chairman and by copy to each member of the Committee his first and second choice nominations for each award and shall provide with his nominations, his appraisal of the qualifications of those he is nominating and his reasons for preferring them. If a member of the Committee feels that no award should be given, he should so state by August 1st. Failure to nominate either a first or second choice for either award will not relieve a committee member of the duty of voting on the nominations made by others, or of voting that no award be made if those nominated are not considered satisfactory.

Between August 1st and September 1st, the Chairman shall arrange for a letter ballot election by the Committee of those to be nominated for the Awards. If no decision can be made by the first ballot, then a second ballot shall be taken on those who have received the most and second most votes on the first ballot. In the event of a tie for first place on the first ballot, only those names shall be considered in a second ballot. Similarly, in the event of a tie for second place, these names, plus the first place name, shall be considered on a second ballot. In the event a person is nominated for both awards, his vote for one award shall not be considered in determining his standing in the vote for the other award.

If the Committee should decide that none of those nominated are worthy of the Awards, or if no nominations are received, the Committee may recommend to the Directors that one or both Awards be withheld for reasons to be stated in their report to the Directors.

Recommendations of the Committee shall be reported by the Chairman of the Committee to the Board of Directors through the Executive Secretary not later than November 1st.

In the event that the Committee should be unable to reach a decision on candidates for either or both awards, the Committee shall provide the Board with a list of all those proposed for consideration and the number of votes received by each on the last ballot of the members of the committee.

The Board of Directors shall then

(Continued on Page 72)

Notes for Guidance Of Awards Committee

A. The Committee shall not recommend the same individual for both awards in any one year.
B. Previous recipient of an award is not eligible to receive the same award again.
C. The recipient of one award is not barred from receiving the other award for some other year.
D. In considering the qualifications of those nominated for awards, the nominee's total contributions should be taken into account and recent activities should not take precedence over the recognized results of earlier work. Other things being equal, awards should be made to the candidates who have been contributing consistently for the longest period.
E. Candidates need not be residents of North America.
F. Candidates need not be members of the National Association of Corrosion Engineers.
G. Awards shall be limited to living persons.
H. Recipients of the Willis Rodney Whitney and the Frank Newman Speller Awards and the recipient or recipients of the Junior Award should be guests at the annual NACE banquet.
I. The President and Vice President of the Association and the Chairman of the Awards Committee should not be considered as candidates for awards.

February 27, 1958

CALGON CORROSION CONTROL PROTECTS THESE VITAL AREAS

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North Central Region News

October 20-22 Conference To Be Held in Cleveland

Registration fee of $12.50 will entitle registrants at the 1959 North Central Region Conference (October 20-22, Statler-Hilton Hotel, Cleveland, Ohio) to attend all technical meetings, symposia, fellowship hour, luncheon and one plant trip.

About 30 technical papers on a wide variety of subjects of interest to corrosion engineers will be presented. A listing of these papers, abstracts, authors' biographical sketches and photographs were printed on page 84-90 in the September issue of CORROSION.

Special entertainment is planned for the fellowship hour on Tuesday, October 20, and the luncheon on October 21.

Registrants will be able to choose from four plant trips scheduled: Heil Process Equipment Company and Pfaudler Corporation, representing materials of design; Diamond Alkali Co., representing protective coatings; East Ohio Gas Co., and Harco Corp., representing cathodic protection; and a general interest tour of Ford Motor Company's engine plant.

●

Detroit Section held its annual L. D. Cook, Jr., Memorial Meeting on September 17. The first meeting of each program year has been set as a memorial to the late Dave Cook, a founding member of the section.

Guest speaker was R. McFarland, Jr., technical director of Hills McCanna Co., Chicago. He spoke on non-metallic materials for corrosive service.

●

Kansas City Section September 14 meeting featured a talk on inspection for corrosion using the audio gauge, given by Wilbur W. Wright of Phillips Petroleum Company.

●

Chicago Section will discuss coal tar epoxy resin coatings at its October 20 meeting to be held in the Chicago Engineers' Club.

At the September 15 meeting Miss Amalia Miller of Underwriters' Laboratories, Inc., was the guest speaker.

Election of section officers will be held in November to be installed on calendar year basis.

Tentative dates and programs for 1959-60 have been planned as follows: November 17: Cathodic Protection; January 19: Refinery; February 16: Corrosion Roundtable Discussion; March 15: Lead Sheath Cable; April 19: Drainage; and May 17: Cost Evaluation.

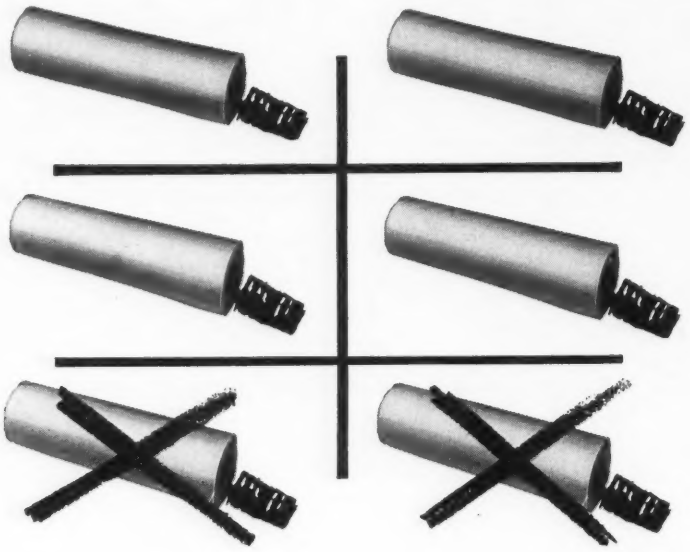
Procedure in Selection—

(Continued From Page 70)

determine, either in a regular meeting, or by letter ballot, returnable not later than December 1st in each year, whether the recommendations of the Awards Committee are to be accepted and the awards made as proposed. If the Board of Directors do not approve the granting of an award or awards, as proposed by the Awards Committee, then such award, or awards, will not be made for the year in question. In the event that the awards committee has been unable to reach a decision on candidates for one or both awards, the Board of Directors shall determine by majority vote whether an award (or awards) shall be made and to whom it (or they) shall be granted.

After the Board of Directors have chosen the recipients of the awards, the President of the Association shall notify the recipients not later than December 15th, and shall invite each of them to be present at the annual meeting or other occasion when the awards are to be presented. The Chairman of the Committee shall arrange to have appropriate certificates inscribed for presentation to the recipients of the awards and on occasion of any public presentation of the awards he shall present the recipients to the President or whoever may be serving in his stead as Chairman of the meeting, to receive the awards at his hands.

The actions of the Awards Committee shall be confidential and confined to consideration only as outlined in the procedure until such time as the President receives acceptance or rejection from the awardees following his notification to them. After receiving acceptance of an award or awardee, the President shall so notify the Executive Secretary who may publicize the information prior to the actual presentation of the award or awards.



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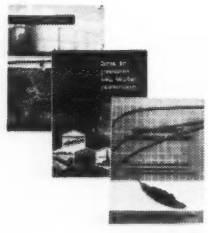

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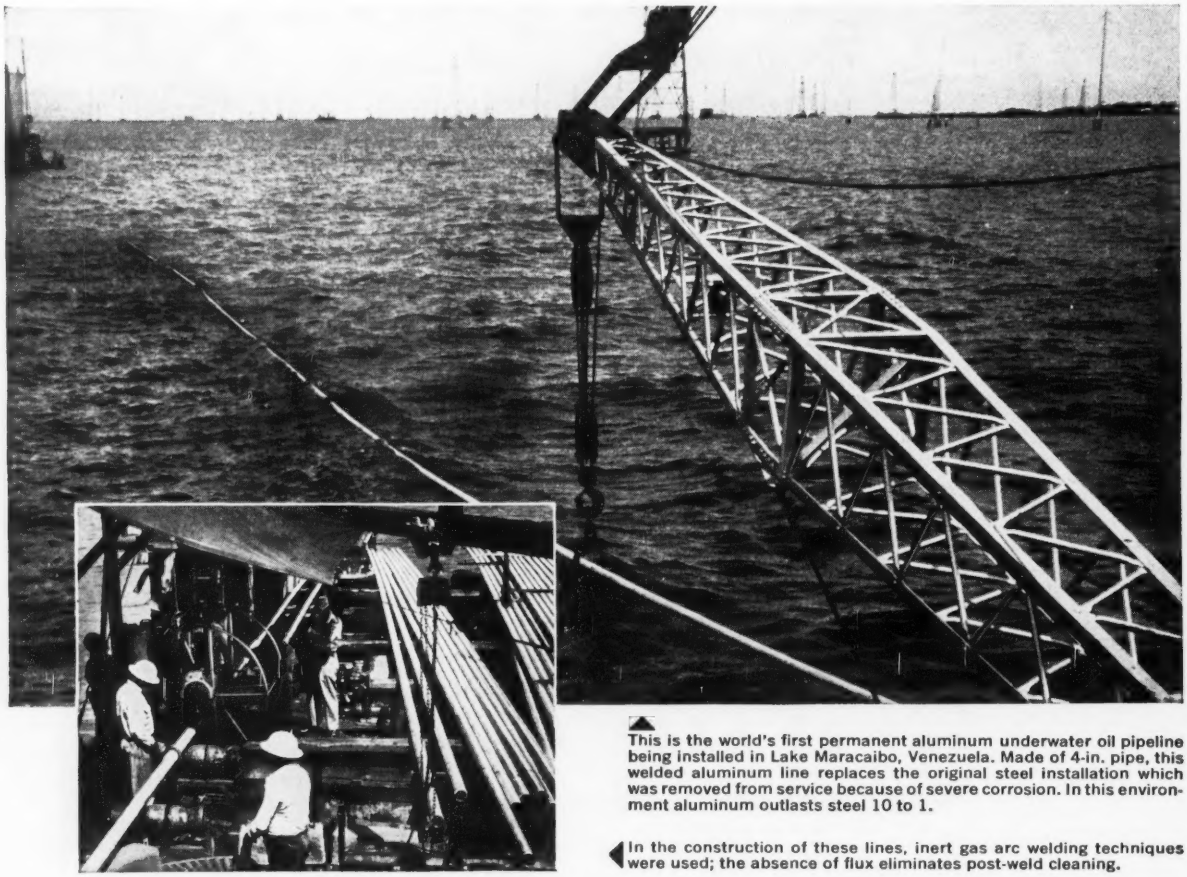
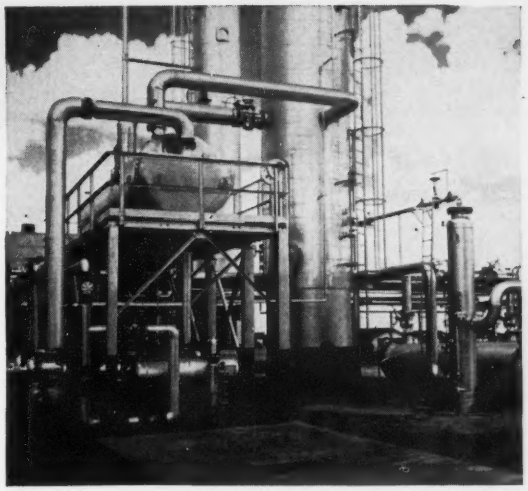
During 1959, ALCOA will conduct engineering conferences in a number of major cities on process industries applications of aluminum. Contact your nearest ALCOA sales office for full particulars and dates.

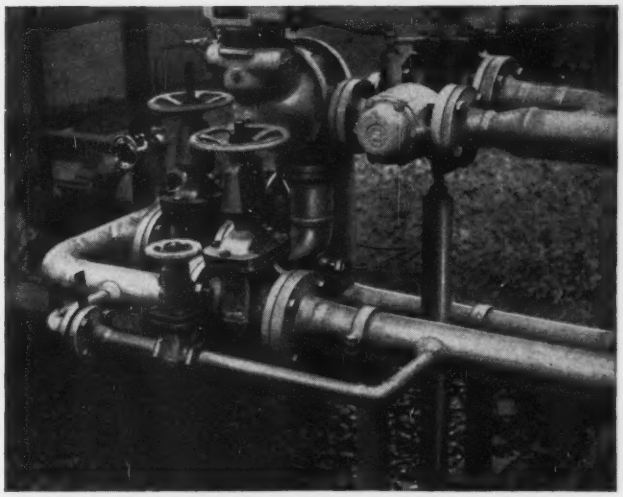


▲ This is the world's first permanent aluminum underwater oil pipeline being installed in Lake Maracaibo, Venezuela. Made of 4-in. pipe, this welded aluminum line replaces the original steel installation which was removed from service because of severe corrosion. In this environment aluminum outlasts steel 10 to 1.

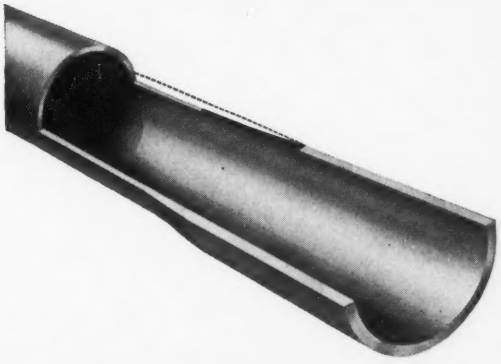
◀ In the construction of these lines, inert gas arc welding techniques were used; the absence of flux eliminates post-weld cleaning.



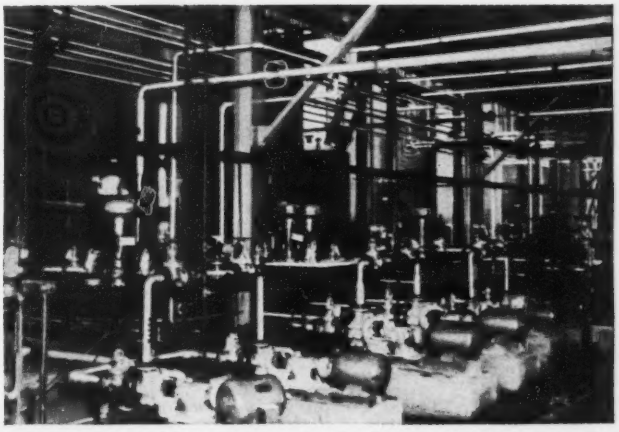
Corrosion and product contamination problems were solved by the use of aluminum in this processing plant. Alcoa Aluminum provides the lowest priced pipe able to resist the action of vinyl acetate and other corrosive liquids such as fuming nitric acid, hydrocyanic acid, acrylonitrile, etc.



Highly resistant to H_2S and CO_2, aluminum pipe has been proven in service in gas purification plants.



Alcoa's new UNISTRENGTH Pipe cuts cost, cuts weight with no sacrifice in bursting strength. The secret: wall thickness is reduced everywhere but at the ends where it is needed for joining. This compensates for the local reduction in strength caused by the heat of welding on popular heat-treatable pipe alloys. Available in a range of sizes with ends matching uniform wall pipe of the same size and ASA schedule number.



Naval store plants find aluminum pipe and Unitrace ideal choices because they do not discolor or downgrade resin and other pine products.

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Please send me the following literature on Alcoa Aluminum for piping, fittings, and many other uses in the process industries.

☐ 10197 Aluminum Pipe and Fittings
☐ 10460 Process Industries Applications of Alcoa Aluminum
☐ 20849 Resistance of Aluminum Alloys to Weathering and Resistance of Aluminum Alloys to Chemically Contaminated Atmospheres
☐ 20272 Aluminum Alloys for Handling High Purity Water
☐ 20268 Resistance of Aluminum Alloys to Fresh Waters
☐ 20935 Designing to Prevent Corrosion
☐ 10415 Welding Alcoa Aluminum

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CUBAN SHORT COURSE plans were begun by the group shown above in Havana. Planned for 1960, the corrosion short course will be held at the University of Havana. From left to right are George Quintana, Compania Cube de Electricidad, William Gundaker, Compania Cuba de Electricidad, H. B. Sasman, Sasman Engineering Co., Miami, Fla., and Sergio Fernandez, Sasman Engineering, Miami.

NATIONAL and REGIONAL MEETINGS and SHORT COURSES

1959

Oct. 5-8—Northeast Region Conference. Lord Baltimore Hotel, Baltimore, Md.

Oct. 12-15—South Central Region Conference. Denver, Colo. Cosmopolitan Hotel.

Oct. 20-22—North Central Region Conference, Cleveland, Statler Hilton Hotel.

1960

January 17-20—Canadian Region Eastern Division. Toronto.

February 10-12—Canadian Region Western Division. Vancouver.

March 14-18—16th Annual Conference and 1960 Corrosion Show. Dallas, Texas, Memorial Auditorium.

Oct. 11-14—Northeast Region Conference. Prichard Hotel, Huntington, W. Va.

Oct. 19-20—North Central Region Conference. Schroeder Hotel, Milwaukee.

Oct. 25-28—South Central Region Conference, Mayo Hotel, Tulsa.

1961

March 13-17—17th Annual Conference and 1961 Corrosion Show, Buffalo, N. Y., Hotel Statler.

Oct. 9-11—North Central Region Conference, St. Louis, Chase Park Plaza Hotel.

Oct. 24-27—South Central Region Conference, Houston, Shamrock Hotel.

Oct. 30-Nov. 2—Northeast Region Conference, New York City, Hotel Statler.

1962

March 18-22—18th Annual Conference and 1962 Corrosion Show. Kansas City, Municipal Auditorium.

October 16-19—South Central Region Conference, Hilton Hotel, San Antonio, Texas.

SHORT COURSES

1959

November 16-20—4th Annual General Florida Conference 1959 Corrosion Short Course. Key Biscayne Hotel, Miami.

December 7-11—University of Illinois Corrosion Control Short Course. Urbana Campus.

1960

January 22-23—5th Annual Houston Section Corrosion Control Short Course. University of Houston.

April 27-29—Portland Section Corrosion Control Short Course.

Southeast Region News

JACKSONVILLE SECTION CHAIRMAN recently elected is Henry T. Rudolf, president of Atlantic Coatings Co., Inc. He has a BS degree from Lafayette College and has been associated with the steel industry in the U.S. and abroad. He was one of the founders of the Jacksonville Section and was its first chairman. Other 1959-60 officers elected are Vice Chairman H. D. Collette, Florida Light & Power Co., and Secretary-Treasurer D. W. Hendrickson, City of Jacksonville.

Rudolf

SECTION CALENDAR

October

5 Central Oklahoma Section. Tropical Cafeteria, Oklahoma City.
5 North Texas Section.
6 Shreveport Section. Captain Shreve Hotel. Cathodic Protection of Pipeline Coatings.
8 West Kansas Section.
13 San Francisco Bay Area Section.
20 Chicago Section. Chicago Engineers Club.
22 Teche Section. Petroleum Club.
22 Edmonton Section. Corona Hotel.
23 Philadelphia Section.
27 East Texas Section. Longview Hotel in Longview.
27 Panhandle Section.
29 Detroit Section. Liquid Corrosion—Theory and Practice. Plant trip to brewery and talk on brewery corrosion by H. Rosenbusch, Jr.

November

2 North Texas Section.
3 Shreveport Section. Capt. Shreve Hotel. Well Corrosion Control.
4 Central Oklahoma Section. Tropical Cafeteria. Oklahoma City.
5 West Kansas Section.
5 Pittsburgh Section. Coatings Roundtable Discussion by five speakers.
10 San Francisco Bay Area Section.
16 Baltimore-Washington Section. Problems, Testing and Mitigation Methods Peculiar to Corrosion Control of Lead Sheath Cables, by T. J. Maitland.
17 Chicago Section. Chicago Engineers Club. Cathodic Protection.
17 San Joaquin Section.
18 Los Angeles Section.
19 Teche Section. Petroleum Club.
19 Detroit Section. Annual election. Uses of Polyester-Fiberglass in Chemical Industry, by W. A. Symanski, Hooker Chemical Corp.
20 Ohio Valley Section. Louisville YMCA.
23 Tulsa Section.
23 Atlanta Section.
24 East Texas Section. Longview Hotel in Longview.
24 Panhandle Section.
26 Edmonton Section. Corona Hotel.

4th Florida Short Course Tentative Program Given

Tentative program schedule for the 4th Annual General Florida Conference 1959 Corrosion Short Course is given below. The course will be held November 16-20 at the Key Biscayne Hotel, Miami, Fla.

Monday, November 16

Moderator: J. B. Prime, Jr., Florida Power & Light Co.
Registration
Aircraft Corrosion
Missile Testing: Paul Jurek, Pan American World Airways

Tuesday, November 17

Moderator: S. Miller, University of Miami
Fundamentals of Cathodic Protection. J. A. Lehman, Electro Rust-Proofing
Fundamentals of Cathodic Protection: B. Husock, Harco Corp.
New Developments in Rectifiers: Harry R. Aschan, Good-All Electric Mfg. Co.
Discussion

Wednesday, November 18

Corrosion of Lead Cable Sheaths: K. G. Compton, Bell Laboratories
Pipe Type Cable: speaker pending
Corrosion Survey Procedure: E. W. Seay, C & P Telephone Co. of Virginia
Pipe Line Corrosion Problems: Marshal E. Parker, consulting engineer
Instrumentation: R. F. Hadley, Sun Pipe Line Co.
Discussion

Thursday, November 19

Moderator: E. J. Tilton, Jr., Florida Power & Light Co.
PVC Pipe: A. H. Henning, General Tire & Rubber Co.
PVC Plastisol Coatings: speaker pending
Condenser Tube Corrosion Problems: Wilson Lynes, Revere Copper & Brass Co.
Urethane Coatings: F. C. Weber, Phelan-Faust Paint Mfg. Co.
Non-Destructive Testing: R. F. Mikus, Pittsburgh Testing Laboratory
Discussion

Friday, November 20

Moderator: H. T. Rudolf, Atlantic Coatings Co.
Molecular Structure and Coating Performance: H. F. Payne, University of Florida
Elastomeric Chemical or Heat Resistant Coatings Based on Neoprene, Hypalon, Fluro Rubbers, Urethances and Silicones: G. K. Volgelsang, Gates Engineering Co.
Guide to Selection of Coal Tar Coatings for Protection: N. T. Shideler, Pittsburgh Chemical Co.
Epoxy Coatings: speaker pending
Coating Developments: Donald W. Fergusson, V. W. Ginsler and W. R. Ahrberg, Rust-o-Leum Co.
Discussion

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GENERAL NEWS

Information Survey Given on Translation Services Available

A survey is given below of information and news story published in CORROSION concerning the availability of abstracts and translations of technical information printed in languages other than English, especially Russian. Persons interested in this field are urged to review these items for the latest available information.

The National Association of Corrosion Engineers does not have copies or translations of the original articles.

Additional information concerning foreign technical information and translation services will be published in CORROSION as it becomes available.

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March: Foreign Technical Information Center Set up by United States Department of Commerce..	85
May: Technical Translations of Soviet Orbit Data Available.....	104
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August: English Abstracts of Foreign Language Articles.....	96
October: Abstracts of Soviet Technical Journals Offered.....	91
November: Periodicals—Current Contents of Chemical Publications	82
1959	**Page**
February: New Publication to List Technical Translations	79
April: Book News—Technical Translations (semi-monthly).....	86

Translation Services

The following list of translation services is published for information only, without warranty by NACE of any kind concerning the organization or their work. All organizations listed provide translations of technical articles for a fee.

Special Libraries Association Translation Center, 31 East 10th St., New York 3, N. Y.

Henry Brutcher, Technical Translations, P. O. Box 157, Altadena, Cal.

Associated Technical Translations, P. O. Box 271, East Orange, N. J.

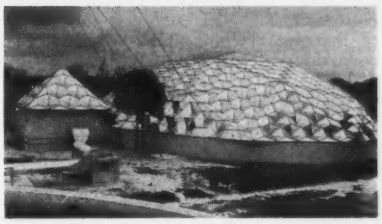
Engineering Societies Library, 29 West 39th Street, New York City.

Copies are available on request of information on Soviet scientific information from National Science Foundation, 1951 Constitution Ave., N.W., Washington 25, D. C.

Nuclear Data Available

"Nuclear Data Sheets" and "Nuclear Theory Index Cards" are available from the National Academy of Sciences, National Research Council, 2101 Constitution Ave., Washington 25, D. C.

The data sheets are a cumulated and continually revised system presenting data on radioactivity and nuclear energy levels. The index cards are a cumulated system for theoretical papers in low energy nuclear physics.



GEODESTIC COVER of hexagonal aluminum panels and stainless steel frames shown above was used at the Sarasota, Fla., sewage treatment plant engineers as a means to suspend a fabric baffle to capture offensive and corrosive hydrogen sulfide. A tent-like polyester fiber covers the 10,000 square foot filtering area and is woven close enough to trap the fumes from the filter bed. Before installation of the fabric baffle, hydrogen sulfide escaped over the entire area of the trickling filter and caused civic complaints and legal actions. Now trapped inside the geodesic cover by the baffle, the fumes are drawn by a suction fan down to filter level and funneled into a 75-foot stack.

Cash Awards to Be Given Top Electroplating Papers

Cash awards will be given for the best paper submitted on the use of electroplating to solve a design, engineering or manufacturing problem or to improve a product.

Five hundred dollars will be given for first prize; $250 awards and five additional prizes of $50 each for the runner-up papers. Competition ends on February 1, 1960.

Additional information can be obtained from Product Finishing Electroplating Industry Award Competition, Gardner Publications, Inc., 431 Main Street, Cincinnati 2, Ohio.

ASM Nov. 2-6 Meeting

NACE member H. H. Uhlig, MIT professor of metallurgy, will present a paper titled "Some Metallurgical Factors Affecting Stress Corrosion Cracking of Austenitic Stainless Steels" at the November 2-6 National Metal Exposition and Congress sponsored by ASM in Chicago, Ill.

Texas Atomic Conference

The Second Annual Texas Conference on Utilization of Atomic Energy will be held November 12-13 on the Texas A & M College campus, College Station. General Alvin J. Luedecke, general manager, AEC, is general chairman of the conference and will be the banquet speaker.

Metallurgical Society

Papers on plastics and ceramics will be included in a symposium at the annual fall meeting of the Metallurgical Society of the American Institute of Mining, Metallurgical and Petroleum Engineers to be held at Chicago's Morrison Hotel on November 2-5.

•

NACE's 16th Annual Conference and 1960 Corrosion Show will be held March 14-18 in the Memorial Auditorium, Dallas, Texas.

California U. to Give Nov. 2-6 Corrosion Control Short Course

A short course on corrosion engineering, sponsored by the University of California Department of Engineering and Engineering Extension, will be held November 2-6 on the Los Angeles campus.

Purpose of the course is to emphasize the problems associated with the high and low humidity change in marine atmosphere of the Pacific Coast region. Fundamentals of corrosion reactions and corrosion control will be reviewed. Day-to-day problems will be related to new developments techniques and research.

Intended for persons with a BS or equivalent experience, the course will be organized to help the recent graduates understand basic principles and to bring the experienced engineer up-to-date with new developments and research trends. Application to practical problems will be emphasized during discussion periods.

Registration fee is $75. Additional information can be obtained from Harold Kayson, coordinator, Engineering Extension, Room 6266, Engineering Bldg. Unit II, University of California, Los Angeles 34.

Attention Librarians

and others who bind CORROSION by Volumes!

The index to CORROSION's 1959 (Volume 15) issues will include alphabetical subject and author references to the articles published in the Technical Topics Section. The growing number and importance of these articles makes necessary to include them in the indexing.

•

Those who bind CORROSION by volumes and who customarily extract Technical Section pages are reminded that it will be desirable to extract also the Technical Topics Section and add it to the Technical Section of each issue. Although the Technical Topics Section does not have cumulative numbering of pages, it will be relatively easy to locate article if this procedure is followed.

Paint-Varnish Meeting

The 37th annual meeting of the Federation of Paint and Varnish Production Clubs will be held October 22-24 in Atlantic City, N. J. A paper of probable interest to corrosion engineers will be "The Bratt Conductivity Cell for Measuring Chemical Resistance."

Analytical Symposium

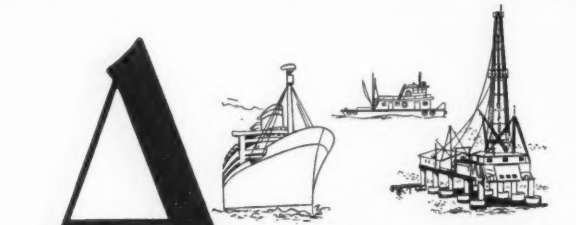
The First Eastern Analytical Symposium is scheduled November 4-6 at New York City's New Yorker Hotel. Topics to be considered include infrared spectroscopy, voltammetry, spectrophotometry, ion-exchange, X-ray spectroscopy and organic analysis.

Coatings Conference

A Coatings Conference sponsored by the University of California Department of Engineering and Engineering Extension Division was held August 31-September 1. Sessions included discussion of instrumentation, film formation and corrosion.

Magnesium Convention

A paper titled "Corrosive Environments and Basic Design Considerations" will be among those papers given at the 15th Annual Magnesium Association Convention to be held October 19-20 at the Hotel Roosevelt in New York City.



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So. San Francisco, Cal., So. Linden Ave., Phone: PLaza 6-1440

PERIODICALS

European Technical Digests. Monthly. In English. Vol. 3, No. 12, December, 1958. 112 pages and cover, 6¼ x 8¼ inches. Organization for European Economic Cooperation, 3, Rue Andre-Pascal, Paris XVI, France. Annual subscription, $12. Six months, $6.

A publication of the European Productivity Agency, an organization of European nations for the purpose of improving productivity. The publication is designed to make available to the world data from about 1000 technical journals and books. Editions are published also in German, Italian, Norwegian, Spanish, Turkish, Croat and Japanese and on a selected basis, Hebrew and Icelandic.

The digests, usually consisting of two to three pages of text and illustrations, are perforated for tearing out. The agency can supply on request photocopies of the original articles digested. The coverage is comprehensive, but attention is given to corrosion as a separate interest.

Technical Translations. 90 pages and cover. Vol. 1, No. 10, May 15, 1959. 7¾ x 10¼ inches. Twice a month. Office of Technical Services, U. S. Dept. of Commerce, Washington 25, D. C. Per annum, domestic $12. Foreign mailings, add $4. Per copy, 60 cents.

Published with public funds in cooperation with the Special Libraries Association Translation Center by the Office of Technical Services. It lists and abstracts translations available from OTS, the Library of Congress, Special Libraries Association, cooperating foreign governments, commercial translators and publishers, universities and other sources.

The references are arranged alphabetically by broad subjects. Book reviews are in a special appendix as are lists of translations in progress, periodicals translated from cover to cover (with price list for translated volumes). There is an alphabetical author index.

BOOK NEWS

Metallkunde II. 179 pages, 4⅛ x 6 inches, paper. (In German) By Heinz Borchers. 1959. Walter de Gruyter & Co., Genthiner Strasse 13, Berlin W 35, Germany. Per copy, German marks, 5.80.

The 1959 edition of this German work on metallography. Liberally illustrated. Alphabetical subject index.

Epoxy Resins. Market Survey and Users' Reference. 200 pages, 8½ x 11 inches, paper. By William N. Bowie, et al. July, 1959. Materials Research, Box 363, Cambridge 39, Mass. Per copy, $18.50.

A compilation of data by a group of graduate students at Harvard Business School during 1958-59. Among the subjects covered are: Description, uses, size of industry, resin producers, formulators, users, general maintenance, flooring, aircraft, automobile, railroads and numerous other industries, future prospects and conclusions.

An extensive appendix with hundreds of references is included. Only one reference to epoxy data published in CORROSION is given.

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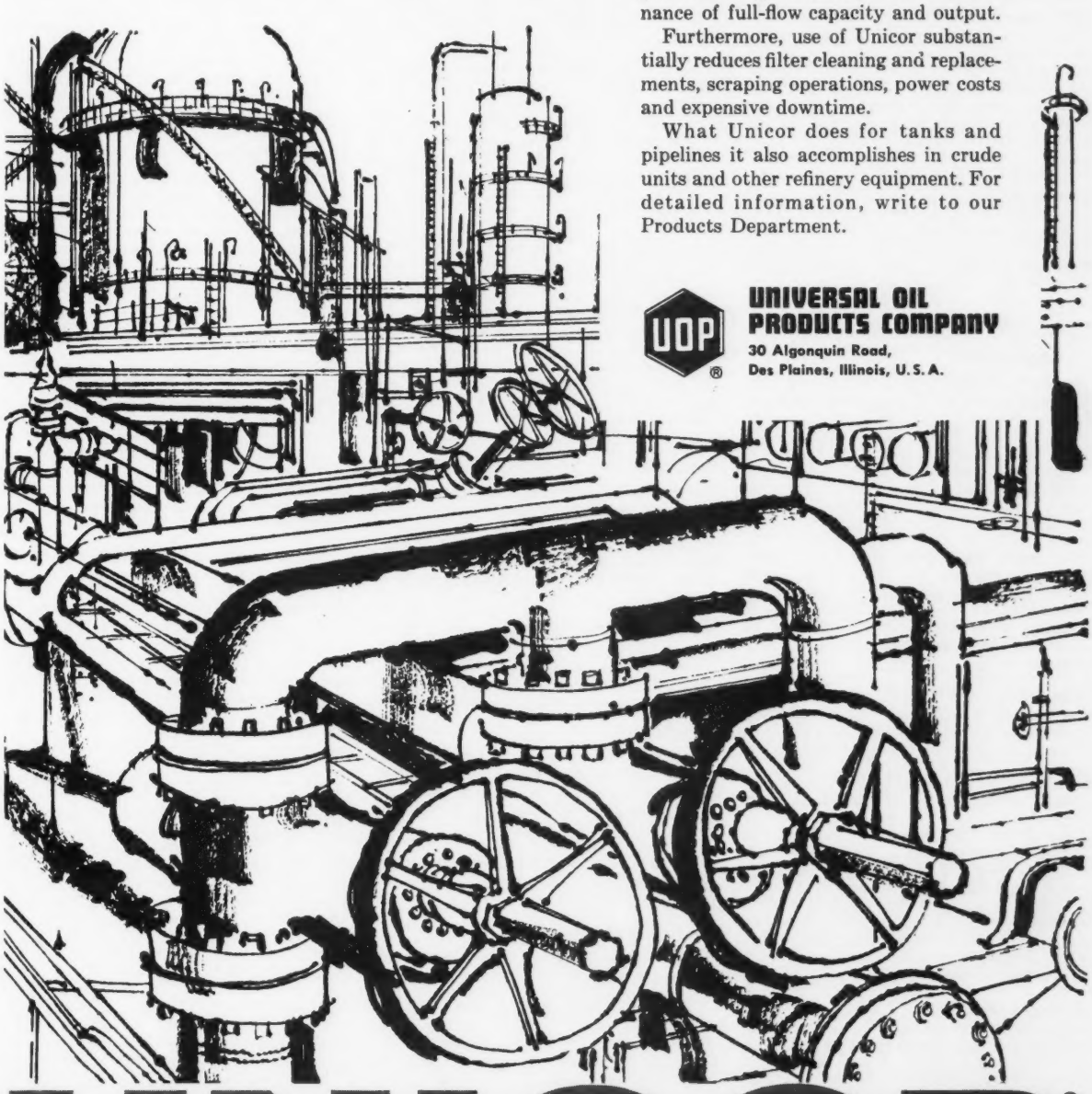

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Equipment Services NEW PRODUCTS Materials Literature

Acidizing

Mud Acid Technical Report is available from Dowell Division of Dow Chemical Co., P. O. Box 536, Tulsa, Okla. Case histories, laboratory data, description, uses and methods of treatment are included.

Batteries

CG Power Packs, potted battery packs with high shock resistance, have been developed by Alkaline Battery Division of Gulton Industries, Inc., 212 Durham Ave., Metuchen, N. J. New design flexibility permits use of any number of the nickel-cadmium button cell batteries to fit different configurations.

Cathodic Protection

Amp-o-Matic, a self-adjusting cathodic protection rectifier for well casings, is being produced by Good-All Electric Mfg. Co., Ogallala, Neb. It eliminates the need to adjust rectifier output when ground bed resistance is changed by heavy rains or long dry periods.

Coatings—Application Equipment

Extension Spray Gun for applying roof coatings and similar heavy materials is being produced by DeVilbiss Co., Toledo 1, Ohio. Designed to handle roof coatings, concrete curing mixtures, foundation coatings, black top sealers, steel tank coatings and sound deadeners, the pole-type gun has a ¼-inch nozzle for a large, round spray pattern.

●

Automatic Metallizing Gun for production, maintenance and research has been developed by SpraRod Corporation, 2795 East 83rd St., Cleveland 4, Ohio. It handles wire sizes from 15 gauge to 3/16-inch in all metals from molybdenum to lead.

●

Quelcor No. 418 is an air dry vinyl coating packaged in an aerosol container for easy application made by Quelcor, Inc., Front and Broomall Streets, Chester, Pa. It has good adherence to most surfaces that have been cleaned and dried. It also can be used as an electrical insulator.

●

New Color Chart No. 5 has been released by Carboline Co., 32 Hanley Ind. Ct., St. Louis 17, Mo. It contains 21 color chips and two new features: chemical resistance of each pigmented color is shown in acid, alkali, oxidizing and weathering environment; and cost percentage higher or lower than standard gray and white is included.

●

Comparative Tests of coal tar epoxy coatings are given in a new brochure available from Amercoat Corporation, 4809 Firestone Blvd., South Gate, Cal. Seventeen laboratory tests are compiled to show behavior of the epoxies when exposed to salts, acids, caustics, bleach, petroleum products, distilled water and weathering. Comparative performances of four coal tar epoxies are described and illustrated for each test.

●

Aluminum Colored Asphalt has been developed by Flintkote Co., 30 Rockefeller Plaza, New York 20, N. Y. Called Aluminized Static, the new coating is recommended for roofs, bridges, water tanks and other exposed surface areas.

●

Alodine 1200S, a new amorphous chromate coating for aluminum, is being produced by Amchem Products, Inc., Ambler, Pa. It is designed to provide corrosion protection and paint bonding qualities for aluminum by chemical means.

●

New Wetting Additive for rust inhibiting red lead paints on steel surfaces is being field tested by the Lead Industries Association, New York, N. Y. The additive is being tested for development of a paint vehicle that will wet the steel as well as or better than linseed oil without the slow-drying characteristics of linseed oil.

●

Large Diameter Pipe coated and wrapped by mechanical means is being processed by Hill-Hubbell & Co., 3091 Mayfield Rd., Cleveland 18, Ohio. Considered the largest diameter pipe to be mechanically coated and wrapped, it is being used as tunnel liner and steel conduit in the Mammoth Pool Power Tunnel Project on the San Joaquin River. Coal tar enamel and primer are used with an asbestos felt wrapper saturated in coal tar.

Engineering Services

Corrosion Engineering Department has been added to the coating and wrapping operations of Rosson-Richards of North Carolina, Inc., 2426 Berryhill R., Charlotte, N. C. Corrosion engineering and construction will be offered; no materials will be sold.

●

Western Cathodic Engineers, 4018 Monty Drive, Box 5286, Midland, Texas, is a new firm offering cathodic protection services, supplies and consulting on estimates and installations.

Inhibitors

Anarust, four new rust-preventive additives and compounds, have been introduced by Techline Division, Wheelabrator Corp., 2400 Ave. V, Vicksburg, Mich. Known as Anarust Type B and G, the additives are used principally in wet blasting abrasive slurry; the compounds are designed for use after the cleaning and finishing process.

Instruments

Alloy 815-R is a new low density resistor material with high electrical resistivity and low temperature coefficient of resistance developed by Hoskins Mfg. Co., 4445 Lawton Ave., Detroit 8, Mich. The alloy is designed for use in manufacturing precision wire wound resistors and potentiometers.

●

Black Light Corporation of America, San Gabriel, Cal., has developed a black light inspection of printed circuits to determine if solder flux, brazing flux and welding slag have been removed. These materials, which can cause corrosion and current leakage, fluoresce under the black light.

Iron

Ductile Iron Pipe production probably will triple this year and reach a total of 100,000 tons annually by 1961, according to estimates made by Ductile Iron Division of International Nickel Company, Inc.

●

Special Report titled "Radiant Heating Slab Construction" is available from A. M. Byers Co., Box 1076, Pittsburgh 30, Pa. It describes installation procedures of interest to architects, engineers and contractors. Sketches show position of wrought iron pipe coils in slab; construction recommendations are also given.

Laboratories

Union Carbide Research Institute is being built at Eastview, New York. Physical and chemical behavior of matter will be studied under ordinary and extreme conditions of pressure and temperature.

Mufflers

JalZinc, zinc coated steel sheets produced by Jones & Laughlin Steel Corp., 3 Gateway Center, Pittsburgh 30, Pa., is being used for the shells of mufflers made by AP Parts Corp., Grand Haven, Mich., for replacement on 1955-1959 automobiles. One reason for use of the zinc coated steel, according to the fabricator, is that its corrosion protective qualities are retained after fabricating operations. AP Parts state that muffler life is increased 25 percent by use of the zinc coated steel.

●

Ceramic Coatings on the inside and outside of automobile mufflers are fused to the steel at high temperatures for shock and corrosion resistance. The coating is also applicable to tail pipes. Passenger car mufflers with the ceramic skin will last about 100,000 miles and cost about the same as present premium quality mufflers, according to Bettinger Corporation, Milford, Mass., developer of the ceramic-on-metal process.

Non-Metallics

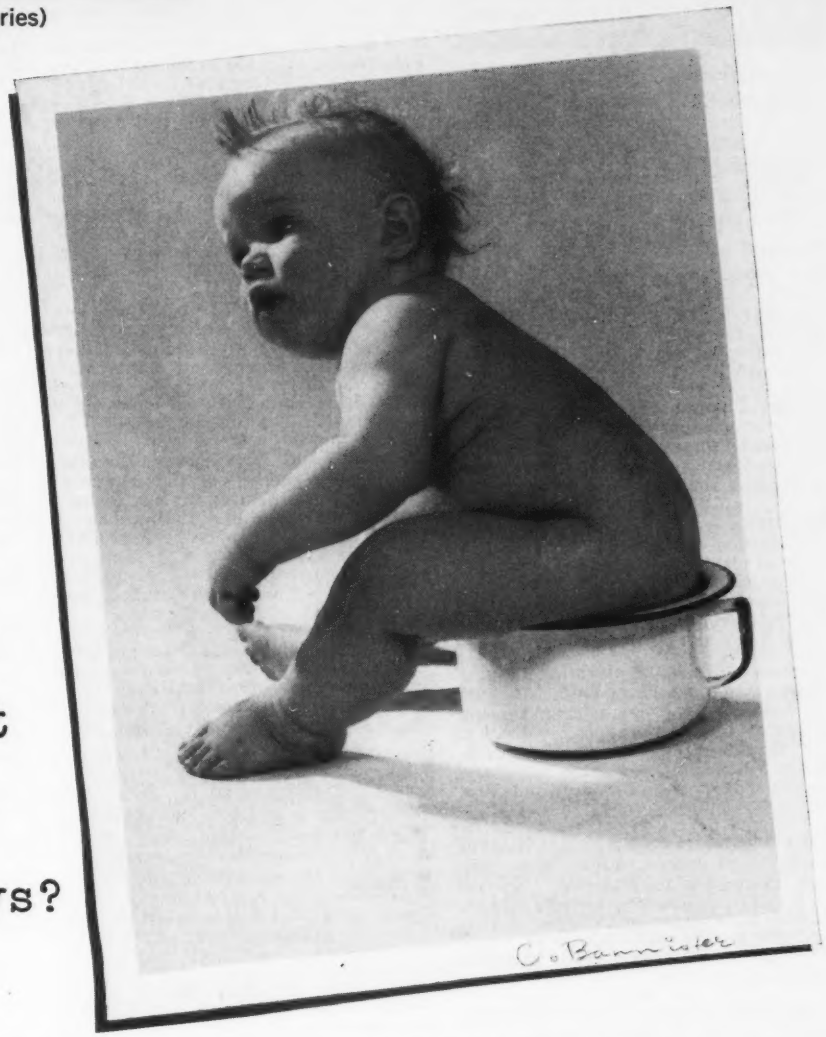
Kofural Impreg is a new chemical resistant material used in fabrication of process equipment. Developed by Wood Preserving Division of Koppers Co., Inc., 719 Koppers Bldg., Pittsburgh 19, Pa., the material is a lumber base impregnated with a monomeric solution of furfuryl alcohol and a catalyst. It can be supplied in special fabricated shapes or in usual lumber forms for fabrication with standard wood-working tools. Resistant to acid and alkali attack, it is also immune to electrolytic or galvanic corrosion.

●

Foamsil, a product of Pittsburgh Corning Corp., 1 Gateway Center, Pittsburgh 22, Pa., is a new lightweight foamed silica material being used in Petro-Chem Development Company's Iso-Flow furnace. Foamsil, a 99 percent pure silica that is resistant to most corrosive chemicals and high temperatures, was used to solve design problems in the furnace caused by weight of other materials.

(Continued on Page 82)

A "PRUFCOAT PRODUCT" PROFILE
(one of a series)



You mean to tell me the pot life of Prufcoat Epoxy 5000 is 2 full days?

Sit tight, friend... because we mean just that! The useful life of Prufcoat "EP" Series 5000—after adding catalyst—is 48 hours. It's one epoxy you can mix today... still use tomorrow. Waste is virtually eliminated. You relax in the knowledge that dollars spent for epoxy coatings won't go down the drain.

Formulated for *both indoor and outdoor* use, polyamide-cured "EP" 5000 coatings exhibit excellent resistance to inorganic acids, alkalies and most all inorganic chemicals. And since they withstand severe organic solvent fume exposure, they are ideal for many complex inorganic and organic corrosive environments.

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Jobs are completed faster. Because with "EP" 5000 coatings, *you just mix and use.* There's no waiting period—no idle waste of time—after the catalyst has been added. Also, recoat time is short. You can apply two coats in one working day. Valuable time is saved.

Yet your "EP" Series 5000 applications—in distinctive stock colors featuring beautiful depth of gloss—need not be restricted because old paints or coatings are already on surfaces you are anxious to protect. An intermediate coat of Prufcoat Primer P-50 eliminates the customary need for complete old paint removal. Then, "EP" 5000 coatings are easily and practically applied.

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NEW PRODUCTS

(Continued From Page 80)

Ultra-Pure Silicon Metal, a material used in the manufacture of transistors and rectifiers, will be produced in a new plant being built by Monsanto Chemical Company's Inorganic Chemicals Division in St. Charles County, Mo., near St. Louis.

Plants

New Research Center being built by Taylor Fibre Co., at Betzwood, near Norristown, Pa., will expand research facilities for study of vulcanized fibre and laminated plastics for use in aircraft, missile and nuclear energy fields.

•

Sodium Chlorate Plant at Aberdeen, Miss., is being enlarged 50 percent by American Potash & Chemical Corporation because of increased consumption by the pulp and paper industry.

Plastics

"Fabrication of Plexiglas," a new handbook available from Arthur Herman, Commercial Plastics & Supply Corp., 630 Broadway, New York 12, N. Y., describes Plexiglas, lists its industrial plant applications and outlines its characteristics.

•

Maintz Caulking Compound, based on chlorosulfonated polyethylene, is being produced by West Chester Chemical Co., Box 39, West Chester, Pa. Available in white and colors, the compound can be applied by gun or knife and cures into a resilient, rubber-like material.

•

Price List and Catalog of materials for fibrous glass reinforced plastics is available from Cadillac Plastic & Chemical Co., 15111 Second Ave., Detroit 3, Mich. Included are ranges of fiber glass fabrics and fibers, polyester and epoxy resins and catalysts, parting agents, pigments and other supplies.

•

Coded Teflon Products are being produced by Pennsylvania Fluorocarbon Co., Inc., 1115 N. 38th St., Philadelphia 4, Pa. Spaghetti, flexible, instrument and heavy walled tubing made from Teflon can be indelibly printed with identification numbers to meet requirements for aviation, electronic, chemical, food, petroleum and general industrial applications.

Technical Topics Included in Index

Technical Topics will be included in CORROSION's annual index. The Topics will be included in the alphabetical subject and author index.

Persons who customarily extract Technical Section pages from each issue for binding are reminded that the Technical Topics pages should be extracted also for a more complete reference to technical information published in CORROSION.

Vicel, a solvent type cement for patching tears and holes in press cloths, centrifuge bags and rotary vacuum filter cloths, has been developed by Technical Fabricators, Inc., 136 Washington Ave., Nutley, N. J. The cement dries and leaves a film of acrylonitrile-vinyl chloride copolymer which has resistance to concentrated acids and alkalies.

•

Garlock Electronic Products is the new name for products formerly sold as Fluorocarbon Products, Inc., a division of United States Gasket Company. Fluorocarbon is now a production department of Garlock Packing Company.

•

Scotchrap No. X-1102 is a new pressure sensitive vinyl pipe protection tape for wrapping straight pipe sections. Made by Minnesota Mining and Manufacturing Co., 900 Bush Ave., St. Paul 6, Minn., the tape can be used for machine applications at any taping angle.

•

Chemically Inert Dip Pipes which withstand severe agitation and thermal shock at temperatures to 500 F have been added to the line of Fluoroflex-T piping components made by Resistoflex Corporation, Roseland, N. J. The new TS dip pipe consists of a Schedule 40 steel housing lined and jacketed with Fluoroflex-T.

•

SRK Semi-Rigid Plastic Pipe, produced by Republic Steel Corporation, was used for drain lines, low pressure air lines, vent stacks and vacuum lines throughout the corporation's new Research Center near Cleveland, Ohio.

•

Ace-Ite, a general purpose rigid plastic pipe, is described in technical bulletin No. CE-80 available from American Hard Rubber Co., Ace Road, Butler, N. J.

Valves

Three-Way Ball Valve made of polyvinyl chloride is being produced by Chemtrol, 10872 Stanford Ave., Lynwood, Cal. Designed to handle corrosive liquids, the valves permit flow in either of two directions or a complete shut-off.

•

Tufline Plug Valves made of stainless steel have been introduced by Continental Manufacturing Co., 247 Park Ave., New York 17, N. Y. The valves are available with four and five ports and have Teflon sleeves which require no lubrication. Specification sheets are available.

MEN in the NEWS

Joseph L. Gillson, chief geologist, E. I. du Pont de Nemours & Co., Wilmington, has been named by the board of directors of the American Institute of Mining, Metallurgical and Petroleum Engineers to represent the International Geological Congress in Copenhagen in 1960.

•

Norman D. Groves has been named assistant manager of research in charge of chemistry and physics for the Carpenter Steel Co., Reading, Pa. A member of NACE, he joined the firm as a corrosion engineer in 1956. In another promotion, **Neil J. Culp** was made assistant manager of research in charge of metallurgy.

Charles L. McCabe has been named head of the Department of Metallurgical Engineering at Carnegie Institute of Technology, Pittsburgh, Pa., replacing **Robert F. Mehl** who will devote more time to research.

•

Edward A. Margus has been made assistant director of product engineering for the Cooper Alloy Corp., Hillside, N. Y., makers of stainless steel castings, valves, fittings and plastic pumps, valves and fittings.

•

Douglas E. Parsons, chief of the Building Technology Division, National Bureau of Standards, Washington, D. C., was elected to honorary membership in the American Society for Testing Materials.

•

Donald G. Sturges has been promoted to associate director of the Research and Development Division, Carborundum Co., Niagara Falls. **Frederick J. Ross, Jr.,** was promoted to manager of the New Products Development Branch. **Wingate A. Lambertson** was made assistant manager of the Engineering Research Branch.

•

Carlisle M. Thacker has joined Taylor Fibre Co., Norristown, Pa., as technical director. He was formerly an engineering consultant with E. I. du Pont de Nemours.

•

Ralph D. Wysong of the Studebaker-Packard Corp., South Bend, Ind., was elected national president of the American Electroplaters' Society, Inc., at the society's meeting last June. He succeeds **Herberth E. Head,** Chrysler Corp., Detroit.

•

Peter D. Shroff has been appointed to the newly-created post of manager, product engineering for NARMCO Resins and Coatings Co., 600 Victoria St., Costa Mesa, Cal.

•

Louis Schiffman has joined the Metalworking Chemicals Division of Amchem Products, Inc., Ambler, Pa. He will do research and product development work.

•

L. W. Roznoy has been appointed director of air and water pollution control for Olin Mathieson Chemical Corporation.

•

David G. Braithwaite has been named head of commercial development of Nalco Chemical Co., 6216 West 66th Place. He is a vice president of the firm.

•

J. Calvin Brantley has been appointed assistant director of research for Union Carbide Nuclear Co., 30 East 42nd St., New York 17, division of Union Carbide Corp.

•

John W. Bruce, Jr. has joined the Barrett Division of Allied Chemical Corp., 40 Rector St., New York 6, as marketing manager, protective products and adhesives.

•

Philip Coddington, general manager of the Carpenter Steel Company's Alloy Tube Division, Union, N. J., retired July 1 after 17 years service with Carpenter. He has been retained as part time consultant to the company. **John S. Kline** has been appointed general manager and chief executive to succeed him.

AMCHEM Information Bulletin

A REVIEW OF PHOSPHATE COATINGS

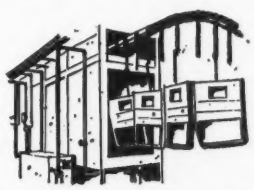
Specified for the Protection of Metal Surfaces

By HUGH GEHMAN, Assistant Manager, Product Development Dept., AMCHEM PRODUCTS, INC.

Phosphate coatings are protective inorganic finishes that actually change the chemical nature of metal surfaces. The metal reacts with the applied phosphate solution to form a nonmetallic, crystalline coating which serves to:

- **Improve paint adhesion**
- **Provide protection against corrosion**
- **Increase lubricity of friction surfaces**
- **Facilitate mechanical deformation of metals**
- **Decorate—in many instances**

Satisfactory protection of steel, zinc and aluminum surfaces against corrosion, paint peeling and blistering,



Typical automotive spray installation.

and hard wear requires precision methods of chemical conversion coating.

Types of Conversion Coatings

There are seven classes of chemical conversion coatings commonly specified and used throughout industry today. They are as follows:

Zinc-iron phosphate (Amchem Granodine). This is the heaviest type of coating (gray in color) used for prepaint treatments on steel, iron and zinc surfaces. The process requires five or six operations: cleaning; rinsing; rust removal, if necessary; coating; rinsing; and a second rinse. Coating weight ranges from 100 to 600 mg per sq. ft.

Medium or large volume production of automobile bodies, appliances, projectiles and cabinets can be handled effectively.

The coating solution improves paint adhesion by forming a crystalline deposit over the metal surface. This deposit is rough, as revealed microscopically, and so offers an ideal gripping surface for paint particles.

Manganese-iron phosphate (Amchem Thermoil-Granodine). This is a heavy black coating used on friction surfaces to prevent galling, scoring and seizing of parts. Typical metal parts treated are pistons, piston rings, gears, cylinder liners, camshafts, tappets and various small arms components.

Typical appliance treatment line.

Iron phosphate (Amchem Duridine). This is a comparatively new process that places a light coating on surfaces for improved paint adhesion. Since cleaning and coating occur in the same bath, it has only three to five stages.

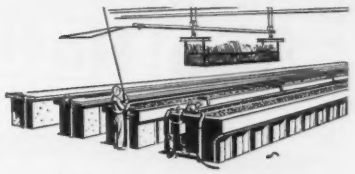
The iron phosphate treatment is a spray process suited for medium to large volume, large or small work. Precleaning is normally unnecessary, an economy factor in its favor.

Products protected by this process are steel or iron fabricated units, such as cabinets, washing machines and refrigerators. Weight of coating is 50 to 100 mg per sq. ft.

Zinc phosphate (Amchem Lithoform). This is a crystalline coating produced on galvanized iron and other zinc surfaces—also cadmium—for improving paint adhesion. The purpose of the coating is to provide a paint-gripping surface and to prevent the reaction between acidic components of the paint and the zinc metal, with the formation of soaps and loss of paint adhesion.

This coating is applied in weights of 75 to 500 mg per sq. ft. There are no limitations on volume or production or on size of products treated. Zinc phosphate coating is used on zinc alloy die castings, zinc or cadmium plated sheet or components, hot dip galvanized stock, and Galvanneal.

Amorphous phosphate (Amchem Alodine). This a protective coating for aluminum and aluminum alloys.

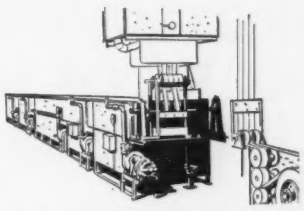


Typical aircraft dip installation.

It may be used in place of anodic deposition for improved paint adhesion and corrosion resistance.

This coating is practical for production in any volume. Coating weight is 100 to 600 mg per sq. ft. Products treated include aluminum awnings, doors and windows, aircraft and aircraft parts, missile parts, roofing and siding. Particularly good when aluminum is painted prior to forming.

Zinc-iron phosphate for oil absorption (Amchem Permadine). This is a relatively heavy coating adapted to the retention of rust-inhibiting drying or nondrying oils and waxes on ferrous metal surfaces. The coating is applied to a weight of 1000 to 4000 mg per sq. ft.



Typical continuous strip line installation.

The process is satisfactory for large or small work in any volume—nuts, bolts, hardware, guns, tools, etc.

Zinc-iron phosphate for metal forming (Amchem Granodraw). This is a specialized coating used in conjunction with a suitable lubricant to facilitate the cold mechanical deformation of steel. The coating acts as an anchor for the lubricant throughout drawing, extrusion, and cold forming operations.

It is a successful treatment for products such as blanks and shells for cold forming, heavy stampings, impact extruded shapes, drawn wire and tube.

For more complete information about any one or all of these chemical conversion coatings, contact an Amchem sales representative or write us at Ambler 32, Pa.

AMCHEM AMCHEM PRODUCTS, INC. (Formerly American Chemical Paint Co.)

AMBLER 32, PA. • Detroit, Mich., St. Joseph, Mo., Niles, Calif., Windsor, Ont.

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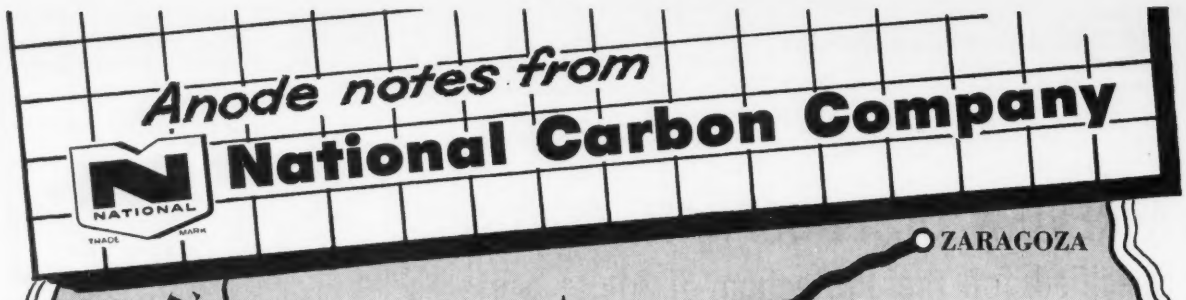

PROTECTION OF 11,000,000 SQ. FT. OF UNDERGROUND STEEL IN SPAIN

"National" graphite anodes for cathodic protection are used around the world. In 1958 four-hundred-sixty 3″x60″ "National" graphite anodes* were installed to protect the 620 mile Spanish J.U.S.M.G. pipeline from Rota to Zaragoza. The 12″, 10″, 8″ and 6″ diameter coated steel pipeline with attendant service pipelines of varying diameters, plus twenty-six tank bottoms total approximately 11,000,000 square feet of steel under cathodic protection.

Because of the wide variations in soil resistivity along the pipeline, each anode and rectifier installation had to be individually engineered. Twenty-six rectifiers with outputs ranging from 20 volts DC to 200 volts DC, from 40 amps to 100 amps were used.

The graphite anodes were placed on 20 foot centers in 12″ diameter x 120″ deep holes surrounded by well tamped coke breeze backfill. The number of anodes in each bed varied, depending upon the current discharged and the soil resistivity. The system is designed to operate for more than ten years.

Wayne Broyles Engineering Company supplied the equipment and installed the cathodic protection system.

UNION CARBIDE

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OFFICES: Atlanta, Chicago, Dallas, Houston, Kansas City, Los Angeles, New York, Pittsburgh, San Francisco • **CANADA:** Union Carbide Canada Limited, Toronto

Abstract

Methods used by Dow Chemical Company at its Freeport, Texas plant to test coating designed for atmospheric exposures and linings used for inside tanks are described. The need for preliminary screening in the laboratory, on location testing in plant environments or in process streams. Among the consideration important in the selection of linings is the availability of technical service from the material suppliers. 2.3.7

A Resume of Procedures for

Testing and Evaluating Chemical Resistant Coatings and Linings*

MOST PROBLEMS and test methods for evaluating coatings for the protection of steel in the plants of the Texas Division of The Dow Chemical Company at Freeport have been described in the literature.[1-5] For those interested in the history of these efforts the references will be instructive. This discussion is confined to a brief resume of test procedures for atmospheric protection and a slightly more detailed account of evaluations of organic chemical resistant linings.

Proximity of the Freeport plant to the Gulf of Mexico and the prevailing onshore winds cause problems common to coastal areas. High humidity contributes to high corrosion rates. The production of chemicals, such as strong acids and strong alkalies, complicates the problem. This is even more true when one of the acids happens to hydrochloric. The higher than average corrosion rates led to calibration of the atmosphere determinined by the corrosion rate of bare steel in all operating plant areas. This method permits projection of test results on coatings to all plant areas.

The evaluation program consists of a series of individual tests, each designed to evaluate a single generic type. Materials for these tests are obtained from numerous manufacturers.

All test materials are applied under carefully controlled conditions and exposed simultaneously as shown in the panel test area seen in Figure 1. Ultimate objective of this program is to arrive at the lowest cost for protection per unit of area per unit of time.

Results of findings after large-scale testing and approval by a paint committee became a part of the plant's paint manual and painting specifications. The constantly repetitive nature of the program permits comparison of current performers with a steady stream of newcomers. In addition special problems of surface preparation, application techniques, film thickness and many other variables are evaluated in a continuous flow of information.

By properly designing experiments data can be evaluated by statistical methods. For example, multiple panel readers and an electronic computer, give accurate and reliable answers to questions for which the tests were designed.

Figure 1—Test panel exposure area.

Poorly designed tests often have resulted in a profusion of data which could not be completely correlated. Conversely, some data has remained unevaluated because of insufficient evidence to produce statistically reliable results.

Because of the aggressive nature of the enviroment, much rust blistering and pitting have been encountered. Additional data regarding primer and possibly top coat selection can be obtained by removing the paint from an exposed panel, as seen in Figure 2 so pit depths can be measured accurately. This leads to definite conclusions in the selection of proprietary paint products.

To the neophyte the number of resin types of coatings available for industrial maintenance painting is bewildering. Dr. L. W. Gleekman, Wyandotte Chemicals Corporation, has published a most interesting paper on this subject entitled, "Classifying Protective Coatings and How to Evaluate the Base Formulation for Maintenance Purposes."[6] He has clearly and concisely described the important resins used in today's maintenance paints or coatings.

Application characteristics of these various types of coatings cannot be evaluated fully at the panel testing stage. These must be evaluated when painting large scale equipment and

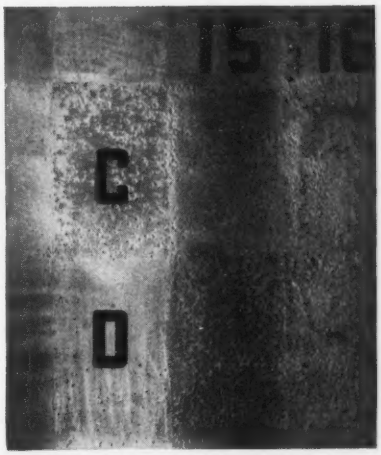

Figure 2—Corroded test panel after removal of coatings.

structures. This is an important phase and should not be omitted. Jumping from the test panel to full scale application is like going from the test tube to tonnage production without the pilot plant.

The interior environments of tanks is readily controlled so results can be obtained in considerably less time.

The selection of chemical resistant lining materials is a problem which faces many process and design engineers. Often the use of linings is overlooked until corrosion or product contamination problems arise in the final stages of pilot plant operations or occasionally in large scale production units. The material of construction is an important variable in process design and in many cases is the controlling variable. Therefore, it is essential that materials should be treated as a process variable as early as possible in any development program.

(Continued on Page 86)

★A paper presented under the title "Test Methods for Chemical Resistant Linings and Coatings,' by R. S. Foster and V. B. Volkening. The Dow Chemical Co., Chemical Engineering Dept., Freeport, Texas at a meeting of NACE Unit Committee T-6A on Organic Coatings and Lining for Resistance to Chemical Corrosion, during the NACE South Central Region Conference, New Orleans, October 20, 1958.

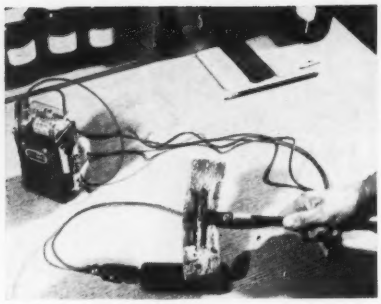

Figure 3—Checking lining test panel with pin hole detector.

Testing and Evaluating—

(Continued From Page 85)

The selection of lining materials is handled in much the same way at Dow as any other engineering problem. Because material of construction is a variable dependent in each case upon a specific process, the selection procedure develops as follows:

1. The aim is to find a material which will withstand the defined conditions and desired limitations of the process involved; therefore, the process must be known in every detail.
2. Once familiar with the process conditions proceed to obtain reliable information on all available materials.
3. Decide which group of available materials is most likely to withstand the defined conditions.
4. Expose the selected group of materials in the laboratory under simulated plant conditions.
5. Expose the best materials from laboratory tests in plant streams and equipment for further evaluation.
6. All available data should be analyzed before the final selection is made. If none of the materials is found to be satisfactory, it is likely that the original process will have to be changed so that less corrosive conditions will be present. Tests are then run under the new conditions.

Following is an example of how this procedure is used.

It is important to recognize the possibilities of a corrosion problem before it actually is experienced. When a corrosion problem is anticipated, first estimate the conditions precisely; define any known limitations and then proceed to select the protective materials which might have promise under these conditions and limitations. Conditions which must be known are:

1) Temperature range of operation
2) Chemical enviroment (pH, polarity of solvents, vapor pressure of mixtures, etc.)
3) Abrasive conditions present
4) Process variations.

Important limitations which must be set are:

1) The allowable cost per square foot of exposed surface,
2) The expected ease of inspection and/or repair,
3) The expected useful life of the process equipment.

With this information in hand, it usually is possible to decide whether to test metal alloys, sheet linings, or organic linings. Usually, if the operating temperatures exceed 300 F, alloys are indicated; if highly abrasive conditions are expected, a thick rubber sheet lining is suggested; and if moderate temperatures and product contamination are the chief considerations, thin film organic linings are indicated.

An article concerning the most generally used linings for process and storage vessels complete with comparative costs is available in the literature.[7] Two NACE unit committee T-6A reports are especially valuable as guides to selection of linings. The first covers vinyl chloride-acetate copolymers[8] and the other epoxy resins.[9] If tank car linings are involved, J. R. Spraul, General American Transportation Corp., has published a good paper on laboratory procedure.[10] Watts[11] discusses coating pipe lines internally in place. Nowacki[12] describes the selection of linings for shipping containers.

For the purpose of this discussion, temperature is assumed to be less than 300 F with no extremely abrasive conditions present. Selection of the material types to be tested is based on a thorough knowledge of the properties of such materials.[8,9] The important properties to consider are the material's resistance to chemicals, solvents, temperature, thermal shock and abrasion. Also, it is important to consider adhesion, permeability and flexibility.

The actual testing of the materials should be carried out in two steps.

(1) The first of these is a laboratory testing stage which consists of the preparation and immersion of small test coupons. For example, evaluations of protective coatings are carried out by initially coating small steel panels and checking them for film defects as shown in Figure 3, and then immersing them in pure chemicals or solvents. This can

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be done by using glass jars as shown in Figure 4. Sometimes it is desirable to evaluate the materials immersed in solvents at elevated temperatures or in mixtures of chemicals and solvents at elevated temperatures. This can be accomplished by using reflux condensers and other standard laboratory heating equipment.[10]

An example of test equipment used at elevated temperatures is given in Figure 5. There is generally a definite interaction between temperature and solvency. A coating which might be usable in ethylene glycol at 100 F, may fail rapidly at 200 F when immersed in this liquid. Test coupons are removed from the solution bottles periodically for microscopic examination. Infrared analysis of the solvents before and after panel exposures can reveal minute amounts of contamination, which might not otherwise be noticeable.

These screening tests serve to eliminate many of the materials which appeared to be usable in the initial considerations. Materials which have survived in the laboratory now should be field tested using the same small coupons inserted in the process streams or equipment. A stock of coupons is maintained in the laboratory for this purpose. These are shown in Figure 6.

(2) The second stage of testing, field evaluation, usually gives reliable information on the durability of various test materials in the environments to which they will be subjected in service. By this time the proper generic type of lining or coating should be clear and only the question of specific manufacturers is left to be determined.

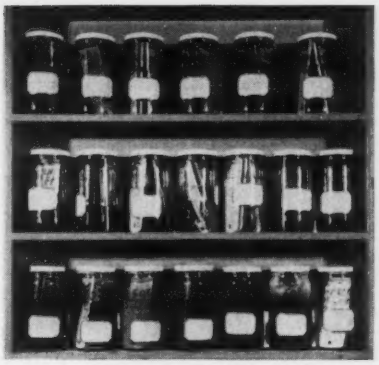


Figure 4—Rack containing lining panels under test at ambient temperature.

Actual selection of a specific lining or coating should now be made based on field test data, estimated applied costs and the anticipated availability of technical service from the supplier of the material chosen.

Experience has shown that there is no short cut to the solution of corrosion problems or the selection of linings. In almost every problem there are two or more materials which have the desired durability. Usually, however, there is a difference either in the application characteristics or over-all cost, but this may not become evident until the first large scale application has been made. Even the most durable materials will fail if they cannot be or are not applied properly.

A few words of caution are necessary in conclusion regarding material costs. Do not buy protective coatings or lin-

(Continued on Page 90)

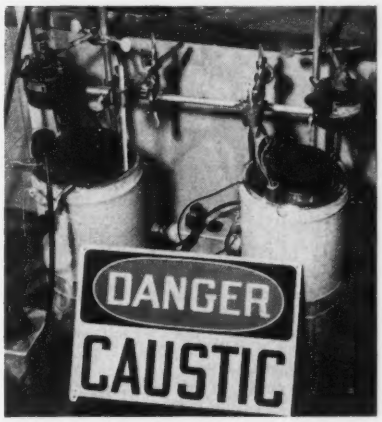


Figure 5—Hot baths containing 50 and 75 percent caustic.

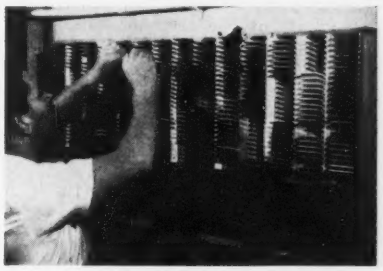
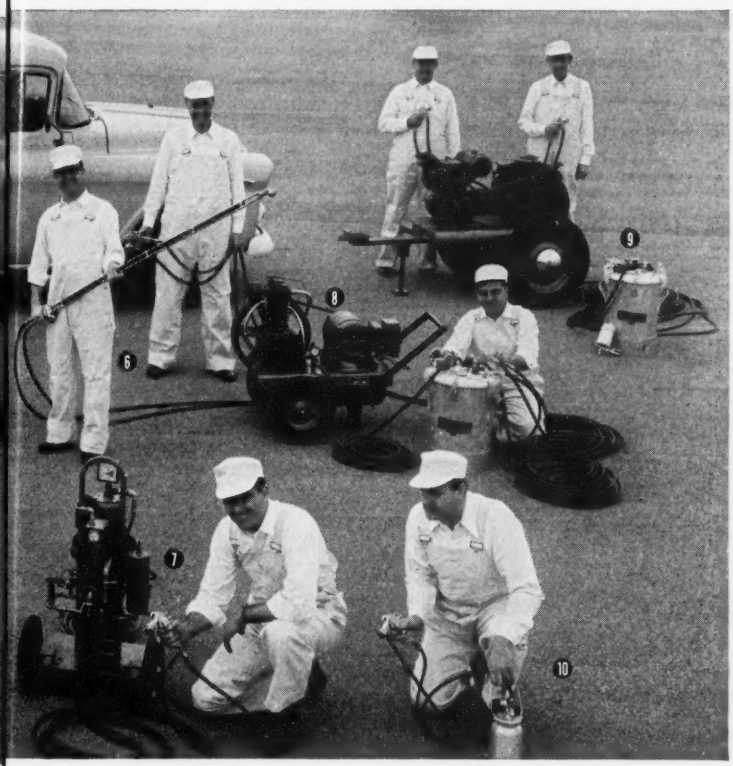


Figure 6—Stock of lining panels ready for testing.



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New Bonding Process Gives Added Versatility to Cladding*

Abstract

Describes a new bonding process for producing clad plates for corrosion resistance. High vacuum, heat and atmospheric pressure are used. The new process is not limited to usual metals used in clad plates and backing plate. Discusses process of manufacture, corrosion tests used, materials available, thickness and size, fabrication and special applications of the cladding. 5.3.4

Introduction

INCREASED OPERATING temperatures, pressures, concentrations and circulation velocities in industries have increased the corrosion agressivness of products stored and process reactions. Clad plated materials have been developed to meet these requirements.

A clad plate consists of a thin layer of relatively expensive corrosion resistant alloy, metallurgically bonded to a strong, economical backing plate such as mild steel or low alloy steel. The heavy backing provides the required strength; the cladding provides the corrosion resistance.

Although some cladding has been done by other processes such as depositing weld metal, continuously bonded cladding is made by only two main processes: by hot rolling pressure and a new patented vacuum bonded type developed by Chicago Bridge and Iron Company at its Birmingham, Alabama, plant. Storage and pressure vessels and other structures that require special metals to withstand corrosive attack are fabricated by this new process.

The patented process bonds metals together without reduction in thickness by a combination of vacuum, heat and atmospheric pressure. It is really a metallurgical union of metals produced in a high vacuum, in a closed envelope. A typical cross section is shown in Figure 1. The evacuated envelope or sandwich is heated in a furnace to a temperature above the melting point of the bonding metal but below the melting temperature of either the clad layer or the backing plate. The sandwich may be assembled to produce from 2 to 10 finished clad plates per heat, the greater number usually being used for thinner plates.

The first production item by this new bonding process was made in 1950, but the process was not developed completely and patented until 1955.

Process of Manufacturer

High vacuum bonding produces a flux-free metallurgical union of the metals. Vacuum is maintained throughout the heating cycle. The outer pressure of about one ton per square foot (from atmospheric pressure of about 14.7 psi) firmly contacts the backing plate, bonding metals and alloy cladding sheets of the evacuated envelope as they soften and relax at the bonding temperature.

The vacuum inside provides freedom from oxidation, removes gases liberated from the surface during heating and promotes wetting of the bonding surface by the molten alloy. Bonding metals can be used that melt just below the bonding temperature and, upon alloying slightly, increase the melting point. Thus, exceptionally strong bonds can be produced that retain their strength at elevated temperatures.

Corrosion Tests

Of interest to corrosion engineers is the accelerated corrosion testing to insure maximum corrosion resistance of the cladding. Finished samples are tested with similar specimens of the "as-received" sheet. Carefully controlled conditions of temperature, agitation, aeration and concentration of corrosive media are maintained. The Strauss test is used also and the familiar boiling nitric acid or Huey test, to guard against grain boundry penetration. Data obtained are used to establish optimum time and temperature cycles, holding time and heating and cooling rates.

Tests in special corrosion media are sometimes performed to duplicate actual service conditions. Results are used in making selection of cladding materials.

Corrosion resistance is the primary objective in cladding processes. Other bonding procedures could produce a slightly stronger bond but would not be considered if corrosion resistance were lessened. Stronger joints are of little concern; the bond shear strength regularly developed is about double the 20,000 psi required by ASTM specifications for clad plates when the cladding is used as part of the design thickness.

Cladding Materials Available

This new bonding process has become a versatile method of combining metals into high quality clad plates. Types of cladding available are listed in Table 1. These cladding types are available in fabricated tanks, pressure vessels and other clad structures. Even the backing plates are not limited to mild or low alloy steels. Stainless steels and other highly alloyed or non-ferrous metals can be used, thus greatly increasing the possible clad plate combinations. Some of the corrosion resistant cladding metals now available are listed below:

A-285 carbon steel plate gives low to intermediate strength; A-201 carbon-silicon steel plate gives intermediate strength; A-212 carbon-silicon steel plate gives high strength; A-204 molybdenum steel plate gives high temperature strength; A-387 chromium-molybdenum steel plate gives high temperature strength; A-203 nickel steel plate is used for low temperature applications; A-202 chromium, manganese, silicon alloy steel plate gives high tensile strength; and A-302 manganese, molybdenum steel plate is produced for high temperature service.

Even gold and platinum, clad on stainless steel, have been produced experimentally, and considerable quantities of pure fine silver on 1-inch solid stainless

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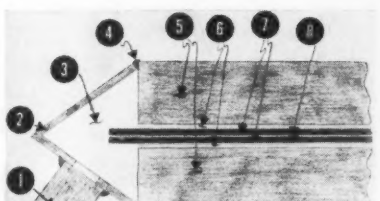


Figure 1—Typical cross section of evacuated envelope or sandwich used to clad materials in new process. (1) Vacuum pipe to pump, (2) Edge seal bar, (3) Evacuated channel, (4) Channel seal weld, (5) Base plates, (6) Bonding material, (7) Cladding sheets and (8) Separating material.

TABLE 1—Types of Cladding

Stainless Steel		Nickel and Nickel Alloys	Other Non-Ferrous Materials
304	321	Nickel	Silver
304L	347	Inconel	Titanium
316	405	Monel	Copper
316L	410	Hastelloy "B"	Cupro-Nickel Alloys
316CB	430	Hastelloy "C"	
318		Hastelloy "F"	
Carpenter 20CB		Nionel	

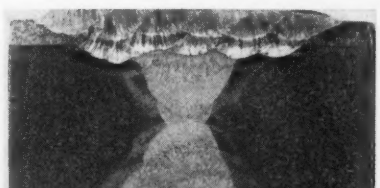


Figure 2—Typical cross section of welded joint.

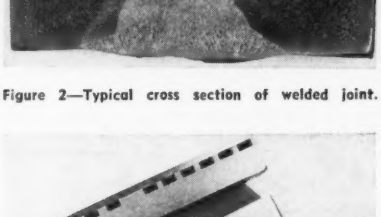
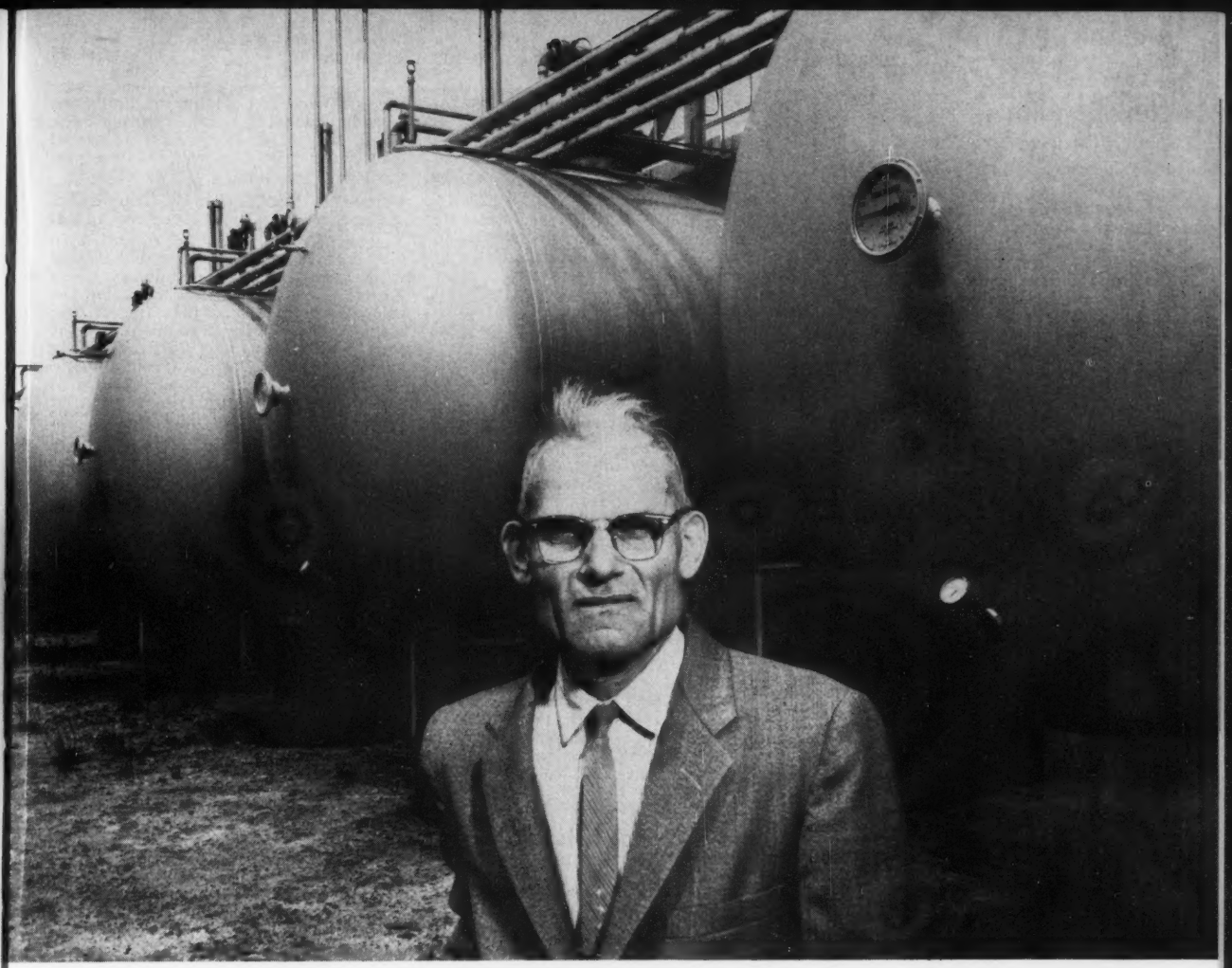


Figure 3—Composite plates with pre-designed internal channels produced by new vacuum bonding process.

★ Extracted from a paper titled "Hortonclad" by R. A. Davis, Chicago Bridge and Iron Co., Birmingham, Ala., presented at a meeting of the 15th Annual Conference, National Association of Corrosion Engineers, March 16-20, 1959, Chicago, Ill.



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New Bonding –

(Continued From Page 88)

have been made into processing pressure vessels.

Other materials for use in cladding are in the experimental or development stage. These include brass, bronze, zirconium, tantalum, molybdenum, Haynes Stellite Alloy X, 25, 6B, multimet and stainless series 200 and 17-7PH (precipitation hardening). Other cladding metals probably will be produced as the need develops.

Thickness and Size

Clad plate is usually limited to approximately 3/16 inch minimum thickness for the backing and 1/16 inch minimum for the cladding, depending upon the material. These are economic limitations: near this thickness range, the cost of bonding approaches the additional cost of purchasing solid corrosion resistant material.

Fabrication

Clad plate can be hot or cold formed into the normal shapes required in the manufacture of large containers and pressure vessels. Hemispherical, ellipsoidal or dished and flanged heads and shell rings regularly produced.

Because of the high temperature bond strength, normal hot forming is performed easily. This strong bond makes service possible in temperatures to 1000 F. Cyclic heating in service has not injured the bond strength, and laboratory tests of alternate heating to 1000 F and quenching for 250 cycles have caused no damage.

Welding the formed plate is easily performed when normal precautions for welding dissimilar metals are observed. Figure 2 shows a typical section of a welded joint.

In composite welds as shown in Figure 2, the abutting edges of the mild steel are beveled to leave an hour-glass shaped gap. This applies to the heavier composite plates and becomes only a single bevel for thinner plates. Stainless cladding is cut back slightly beyond the mild steel bevel. Mild steel is then welded half-way through from one side, and the remainder of the weld is applied from the opposite side. When the welds are to be examined by radiography, they are checked for soundness by X-ray machines before the stainless overlay is applied so that any necessary repairs can be made before the alloy weld is made. Additional X-ray examination is made after the clad is welded, if the cladding is included in the design thickness.

Special Applications

The vacuum bonding process makes production possible of composite plates with pre-designed internal channels, shown in Figure 3. These channels can be of various shapes, and their passageways can be quite intricate if desired. These channeled plates are used in heat exchanger plates, press platens and internally cooled wind tunnel plates.

Regular clad plates and channeled plates can be made with both sides of the backing plate bonded with the same or different materials. An application of this is used in partitioned vessels where the corrosive attack on opposite sides of the plate may result from different media. An unusual application is stainless facing plates for corrosion resistance bonded to the main plate body of copper for heat conduction.

DISCUSSIONS

Comments by George T. Dexter, Seattle, Wa.:

Is this new clad material produced only for fabricating by Chicago Bridge and Iron: If so, is it contemplated that it may be available in the future to other fabricators? How does the surface finish compare with that of conventional stainless clad?

Reply by R. A. Davis:

No, and it is not contemplated that the material will be made available to other fabricators.

Surface finish is the same as on any other cladding and is preserved by the vacuum in the same condition as received from the mill. As on any other cladding, a higher polish could be made afterward if desired.

Comment by John Tom Firestone, Everett, Wa.:

In the case of 300 series of stainless on mild steel, how do you avoid carbon pick up from the mild steel when welding clad sheets together?

Reply by R. A. Davis:

There is no question of carbon pick up when welding the clad sheets together. These are welded, then trimmed and ground flush into sheets as large as the finished plate before cladding. The composition of the weld would normally be approximately the same as the cladding sheets themselves.

Comments by Herbert G. Vore, Nashua, N. H.:

Can you titanium clad plate be satisfactorily welded?

Reply by R. A. Davis:

So far, only two titanium clad pressure vessels have been built. Since the art of making a satisfactory titanium-to-steel weld has not been perfected, pure titanium cover straps were used to close over the seams on the inside.

Comments by Harry Gilman, General Aniline & Film Co., Linden, N. J.:

Have you been successful in forming and fabricating large heating coils from pipe sizes in 2-inch, schedule 40 or 80, approximately 8 to 12 feet in diameter, where the alloys are bimetal such as monel on the inside, stainless on the outside?

In a large pressure vessel fabricated from inconel-clad for caustic fusion service (with chlorides present), the experience in the field indicates that in weld repairing a vessel already installed and already used, that the weld metal rarely has any similar corrosion properties as the base inconel clad? In other words, field welding inconel clad with 80-20 and other recommended welding rods, have continually indicated cracks and corrosion in the weld metal. Have you any solution for having the properties of weld metal and base inconel-clad behave the same after field welding?

Reply by R. A. Davis:

No coils, nor piping as small as 2 inches has been made as this size is apparently too small to be practical. Some piping and even elbows in sizes usually larger than 8 to 12 inches have been made.

No difficulties have been experienced in the welds of inconel clad. Generally, the 80-20 rod is used for the first pass, regular inconel for subsequent passes. So far as we know, welds in inconel clad paper mill digesters have performed very well.

Comments by Charles G. Gribble, Jr., Metal Goods Corp., Houston, Texas:

You mentioned that the sandwich in manufacture is raised to around 2000 F which would take the stainless alloys into solution. This is correct. I had the impression however that the cooling would bring the stainless down into the sensitizing range under 1600 F, cooling slowly so as to cause carbide precipitation. If you repeat and quench, it seems that you would damage your bond. How do you handle this problem?

Reply by R. A. Davis:

Sensitizing of stainless steel cladding has not been troublesome, probably because it has been avoided. Wherever sensitizing might be a problem, low carbon grades of austenitic stainless have been used. Whatever, if any, tendency toward carbide precipitation might remain is greatly eliminated, wherever desirable, by accelerated air blast cooling. A continuing study of best heating and cooling rates provide information for the optimum corrosion resistance of the finished product.

Testing and Evaluating –

(Continued From Page 87)

ings with the price per gallon as the only criterion. The price per mil square foot is a method for obtaining 'comparable cost' for materials. This may be obtained if two things are known about a product: (1) Cost per gallon and (2) percent solids by volume.

Nomographs[13, 14] have been published which by simple alignment of the two known permits direct reading of the comparable cost.

Two precautions are necessary in this regard: (1) 'Comparable costs' are not applied costs; (2) comparisons of costs should be made only after durability is proved.

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Operator checks Ni-Resist pump at Pennsalt Chemical's Portland plant. This particular unit was manufactured by A. R. Wilfley & Sons, Inc. of Denver, Colorado, and pumps caustic at a concentration of 50%. The Duriron Company, Inc. of Dayton, Ohio, also supplied Ni-Resist pumps for the plant.

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Figure 1—Group of nitride-bonded silicon carbide shapes. Many of these parts are subjected to corrosive action during service.

Potentialities and Applications of

Special Corrosion Resistant Refractories*

Abstract

Severe corrosion conditions coupled with abrasion, erosion and moderate to high temperatures create demands for special refractories having properties that make them suitable substitutes for metals in selected applications. Characteristics of special refractories and current applications are discussed. Emphasizes their metal-like properties of thermal conductivity, hot compressive strength and abrasion resistance. Also explains how products can resist extreme conditions of tension, thermal shock and impact by redesign. 6.6.6

Introduction

ORDINARY REFRACTORIES made of minerals such as fireclay, diaspore, kyanite, silica, chrome and magnesia are designed for applications ranging from industrial heat-treatment furnaces to huge open hearths capable of resisting or containing moderate to high heat.

Special refractories, on the other hand, are fabricated of materials made from electric-furnace crystals such as silicon carbide, fused-aluminum oxide and electric furnace mullite. These are crushed, graded, bonded and fired at high temperatures into refractory products. For silicon carbide crystals, the bond can be the silicate type or silicon nitride, a recent development.

A group of nitride-bonded silicon carbide shapes are shown in Figure 1.

Electric furnace melts poured directly into composition or graphite moulds and later annealed are called fused-cast refractories.

Both special and fused-cast refractories retain to maximum degree the properties of their electric furnace crystals and resist higher temperatures than other refractories. However, thermal conductivity, chemical inertness, abrasion resistance, heat-shock resistance, etc., often are more important factors than high melting points.

★ Revision of a paper titled "Corrosion Resistant Special Refractories—Their Applications and Potentialities," by Roy W. Brown and H. G. Noble, Carborundum Co., Perth Amboy, N. J., presented at a meeting of the 15th Annual Conference, National Association of Corrosion Engineers, March 16-20, 1959, Chicago, Ill.

In this article, corrosion resistance of these special refractories will be considered from three points:

1. Substitutes for metals and other materials in applications involving corrosion coupled with abrasion, erosion or heat. Corrosive agents may be gases, slags, bases, acids and molten metals.
2. Product characteristics, their relation to other materials of construction and their use in specific applications.
3. Possible compromises to minimize negative aspects while taking advantage of positive benefits based on tests and field experiences.

Corrosion by Gases

High temperature steam orifices in catalytic crackers made of silicon-nitride-bonded silicon carbide showed little wear after one year's service compared with 2 to 4 months service for stainless steel. This comparison is shown in Figure 2.

In cooling hot SO_2, for example, clay-bonded silicon carbide nozzles spray 15 percent water solution of gas at 150 F into a chamber containing SO_2 gas at 1700 to 1800 F. These nozzles, in use for over 5 years, showed little deterioration. Several were broken mechanically in making arch repairs. Metal nozzles under the same conditions gave less than 2 years' service.

Molecular hydrogen is relatively non-corrosive toward refractories. Above 2400 to 2500 F, however, it dissociates to extremely corrosive nascent hydrogen which reacts with refractories containing silica. For this high temperature service, pure alumina refractories made from electrically-fused Bayer process alumina are used to eliminate silica.

Gas generators processing natural gas, steam and oxygen at very high temperatures produce carbon monoxide and nascent hydrogen synthesis gas. Here the problem is to contain this corrosive mixture in an inert refractory lining that has low thermal conductivity. Bubble alumina insulating brick, made by a process that involves blowing air against a stream of molten aluminum oxide, fills both these requirements. The small hollow spheres form the grog for refractories and castables which combine the inertness and refractoriness of pure fused alumina with the effective insulating action of the hollow spheres.

In HCN generators, nitride-bonded silicon carbide bars are used as supports. Certain metals fill thermal shock and strength requirements but prove inadequate under temperature and corrosive gas conditions. Addition of clay-bonded silicon carbide inner liners extended the service life of nickel tubes used for containing chlorine gas, chlorinated hydrocarbons and hydrochloric acid at 1100 F.

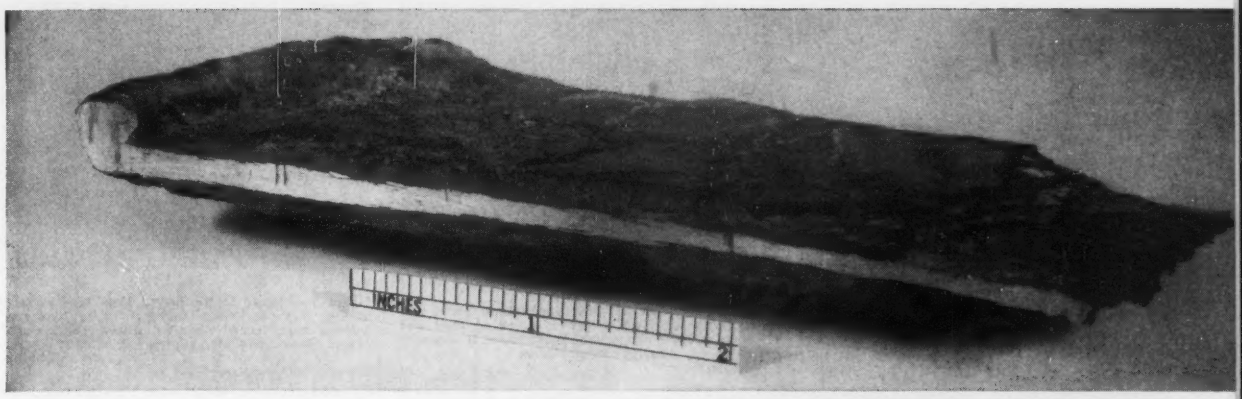
Corrosion by Molten Slags and Salts

In the electrolytic cell producing magnesium metal from molten magnesium chloride, a fused-cast, beta-alumina refractory proved effective at the metal line. Here cast iron is attacked seriously.

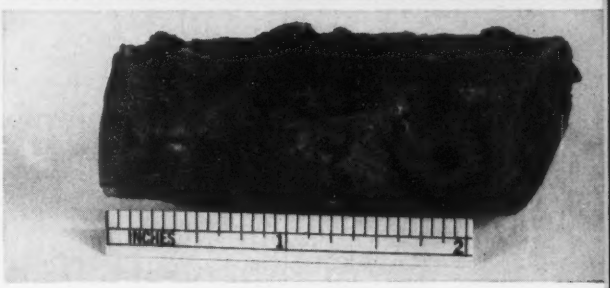
The aluminum reduction cell is a similar application. Aluminum oxide is dissolved in an electrolyte of molten cryolite. Fused-cast refractories and most other ceramics are unusually susceptible to molten cryolite. Both nitride and clay-bonded silicon carbide, however, prove effective. Their high heat conductivity causes formation of a protective frozen cryolite facing. Temporary loss of this facing during thermal fluctuations in the cell does not harm the silicon carbide refractories if the molten electrolyte refreezes in a reasonable time.

In synthesis gas generators handling pulverized coal, steam and oxygen, fused-cast alumina refractories resist the cutting action of the coal ash. These refractories are the accepted linings for glass furnaces, sodium-silicate tanks and rock wool reverberatory furnaces where exceptionally severe corrosion conditions exist. Interior of a glass-melting tank equipped with fused-cast refractories is shown in Figure 3.

(Continued on Page 94)



Wrought Iron plate cut by hand from fire curtain of Marina Yacht Harbor Pier, San Francisco, California—The dense and compact nature of Wrought Iron's protective scale is clearly evident. Its adherence is permanent: on plate after 26 years, and on tubular goods after 73 years of salt water service.



Steel tie rod after cleaning and measuring adjacent to deck where paint was still intact—Note chipped and spalled appearance of scale on rimmed steel tie rod. This tie rod was originally 1" material. When cleaned of rust, the rod measured 0.481", 0.490" and 0.697" on three different diameters.

73 years' exposure to salty sea water produced only 3 mils/year maximum pit depth on Wrought Iron

A structural inspection of the Marina Yacht Harbor Piers in San Francisco, Calif., revealed once again, the decided superiority of Wrought Iron over steel in salt water environment.

Maximum pit depth on Wrought Iron after 73 years' exposure to sea water and sea air was 210 mils: approximately 3 mils/year.

Steel similarly located, showed perforation of ¼" plate after 18 years: approximately 14 mils/year. Severance of one-inch steel rods occurred in 26 years.

No case of Wrought Iron perforation was evident. Either in ¼" plate over a period of 26 years, or in tubular goods over a period of 73 years.

Corrosion researchers reported the following detailed findings in their structural inspection:

- Scale on Wrought Iron was heavy and dense. Scale adhered so tightly that a diamond pattern hammer was required to remove it. Scale on steel was equally thick. But it was so friable that light hammering was sufficient to remove it.
- Some scabbing occurred on Wrought Iron at mean high tide (MHT). But, in every case where a scab was hammered off, a dry inactive pit was found.
- At MHT, the maximum pit depth of Wrought Iron was 210 mils. Steel at the same position showed depths of as much as 360 mils. This occurred even though the steel had been in service less than 26 years. Wrought Iron had already withstood 73 years' service.
- In the area of MHT, where the maximum Wrought Iron pit depth was 210 mils in 73 years, the ¼" (250 mils) steel plate had perforated in less than 18 years.
- Wrought Iron, ¼" thick, adjacent to the ¼" steel plate which had perforated in 18 years, showed a maximum loss of 187 mils in 26 years. This occurred in spite of the fact that the Wrought Iron had been subjected to extreme crevice corrosion not obtaining on the steel.
- One-inch steel tie rods passing through MHT showed reduction in diameter ranging from 293 mils to one inch. This would give pit depths (corrosion from one side only) of from 147 to 500 mils in 26 years. This compares with Wrought Iron pits of 210 mils in 73 years.
- No plausible arguments involving small amounts of copper and nickel can be used to invalidate these data. The compositions of the steel and Wrought Iron are very similar for these elements.

The most casual observation reveals use of two different materials in these docks. The Wrought Iron, after 73 years' exposure to salt water and salt air, is today in much better condition than the steel installed 26 years ago.

A complete inspection report of the Marina Yacht Harbor Piers is available on request. Write A. M. Byers Company, Clark Building, Pittsburgh 22, Pa.

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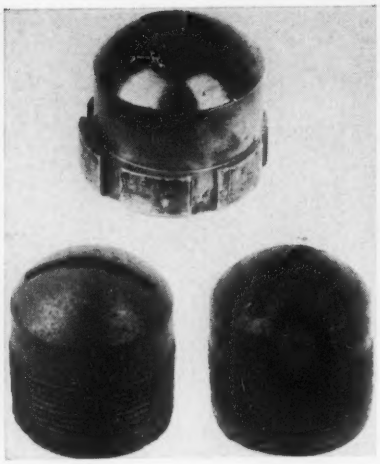

Figure 2—Stainless steel nozzle (top) after less than four months' service jetting high temperature steam in catalytic cracking operation. Nitride-bonded silicon carbide, used under same conditions for one year, is shown at lower right. New part of same material at lower left shows how little wear occurred with the carbide nozzle.

Special Corrosion—

(Continued From Page 92)

Bonded fused alumina refractories have been used as porcelain enamel frit furnace linings because of their resistance to attack by molten enamel.

Slags developed in gray-iron cupolas and in steel mill blast furnaces are corrosive to most materials. Clay-bonded silicon carbide and electric furnace mullite have given satisfactory service for slag hole blocks, continuous front slagging arrangements and blast furnace runner skims.

Incinerators handling corrosive wastes employ electric furnace mullite and silicon carbide refractories as structural components and combustion screens.

Corrosion by Alkalies

Refractories are affected by the same rules that prevail in the chemical industry. Acids resist acids and react with bases and vice versa; reaction rates usually increase with temperature.

Efforts to minimize the weaknesses of the older clay-type bond have resulted in nitride-bonded silicon carbide and fused-cast refractories. They also have provided a relatively new product, self-bonded silicon carbide. This has a density 96 percent that of the true SiC crystal; silicate-bonded silicon carbide runs slightly under 80 percent.

Self-bonded silicon carbide separators in sulfate digesters at paper plants have replaced alloy components that normally

Figure 3—Interior of glass tank using fused-cast refractories to offset corrosive action of melt and to provide longer campaigns. These products minimize glass contamination.

give from 6 to 12 months service. The refractory material has demonstrated that it is at least equal to the metal parts, but in no case has ultimate life as yet been attained. Current condition of the carbide parts show virtually no wear and give indication of lasting for several years. Nitride-bonded silicon carbide separators in sulfite digesters usually outlast alloy by at least four times.

Corrosion by Acids

Special refractories used with acid applications are given in Table 1. Figure 5 shows silicate-bonded silicon carbide wear blocks in a pickling tank using 15 to 20 percent sulfuric acid.

Corrosion by Molten Metals

Special refractories are used for protection and replacement of metals rather than for containment. Most molten metals are contained for long periods with relatively inexpensive materials such as fireclay.

For some aluminizing operations, the submerged heating tube is produced in nitride-bonded silicon carbide, which is resistant to molten aluminum. Thus, the refractory can be used also as sinker rolls for aluminizing sheet and wire, piston and impeller type aluminum pump components, tap-out blocks, floats, troughs, metering devices, pyrometer tubes, etc. Unlike alloys, the refractory does not contaminate the metal.

Severe corrosion in the Pidgeon process for making magnesium is controlled by liners of fused-aluminum oxide and nitride-bonded silicon carbide inside metal retorts. The latter is required because of vacuum requirements.

Properties of Special Refractories

Most important properties of special refractories are thermal conductivity, corrosion resistance, hot compressive strength, hot modulus of rupture, abrasion resistance, heat-shock resistance and permeability. Resistance to elevated temperatures is less important because it is seldom the reason for selecting a refractory.

Thermal Conductivity

Thermal conductivity values of special refractories with those of other materials used for structural purposes are compared in Figure 6. The value for silicon carbide is about 70 percent that of chrome-nickel steels: the safe use limit for prolonged use for stainless steel is about 1500 F; silicon carbide can be used above 2800 F.

(Continued on Page 96)

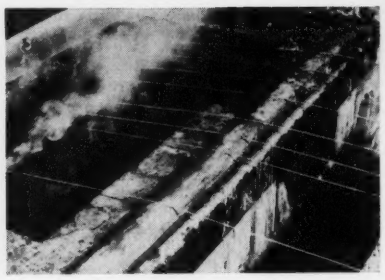

Figure 5—Despite lightly scaled steel wire traveling at 100 feet per minute and hot 15 to 20 percent sulfuric acid, ceramic-bonded silicon carbide wear blocks show little effect after six months' service.

TABLE 1—Special Refractories Used With Acid Applications

Equipment	Refractory
Acid-slurry pump parts	Nitride-bonded silicon carbide
Valves and dampers in sulphuric acid gas lines	Silicate-bonded silicon carbide
Weir blocks in pickling tanks	Fused-cast refractory
Lining water cooled hydrogen chloride burners	Silicate-bonded silicon carbide
Domes in Monsanto Ross-Wilde decomposers handling alkalation sludges	
Radiating domes and rabble blades (see Fig. 4) in multiple hearth roasters	
Mannheim muriatic furnace domes	
Chemical waste incinerators	
Wire guides and sinkers in electrolytic galvanizing (pickling) lines	

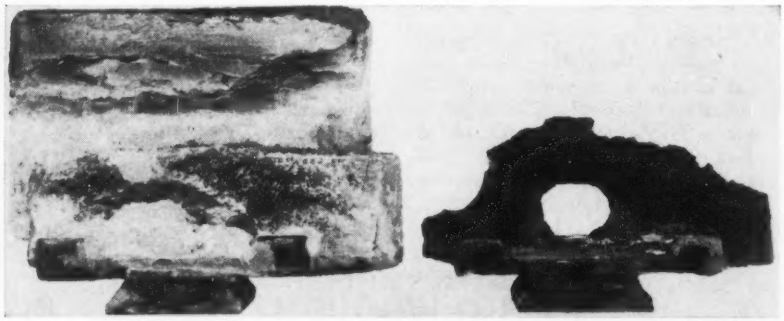

Figure 4—Comparison of steel (shown at right) and silicon carbide (at left) rabble blades after six months' service in the same furnace.

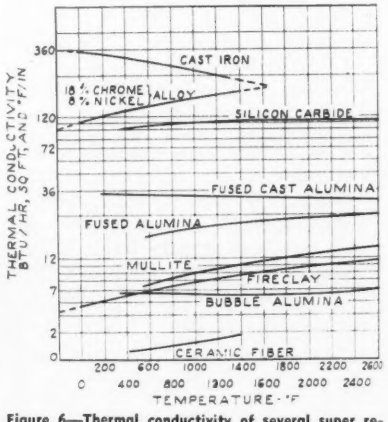

Figure 6—Thermal conductivity of several super refractories compared with other materials of construction.

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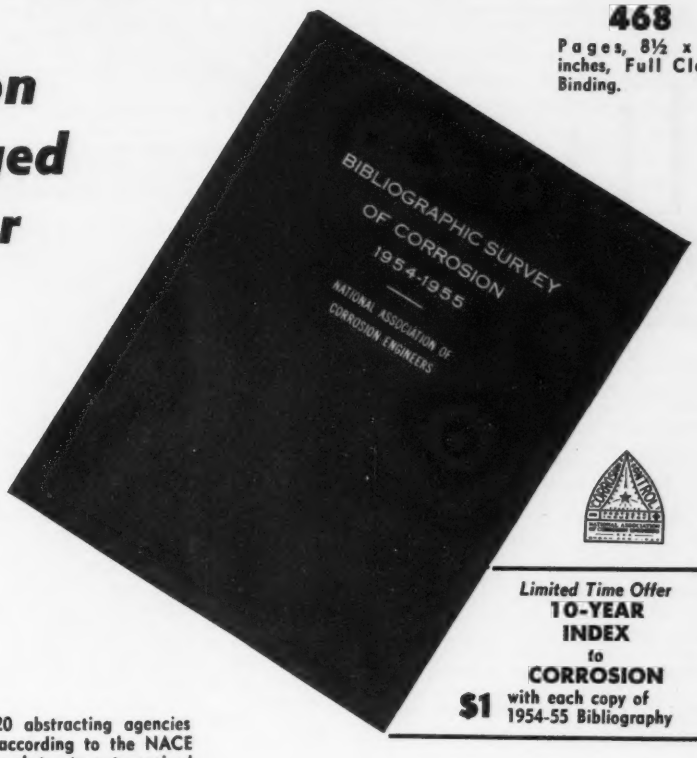

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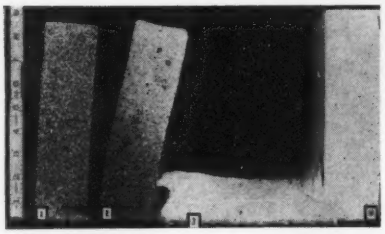

Figure 7—Electric furnace mullite brick (M) shows almost no contraction after 5 hours at 3000 F. The other brick, commonly used to resist heat, are in various stages of deformation.

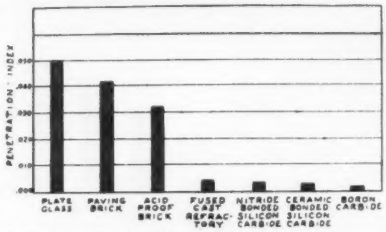

Figure 8—Results of Zeiss impingement tests on non-metallics.

Special Corrosion—

(Continued From Page 94)

In many instances, heat transmission transcends corrosion resistance. Two examples are a submerged silicon carbide heating tube in melting aluminum and a pyrometer protection tube immersed in aluminum for closer temperature control. Without rapid heat flow, the corrosion resistance of the material would be of little value.

If the silicon carbide radiating dome in a Mannheim furnace were replaced with a fireclay dome, a 2000 F heat boost in the combustion zone would be necessary to maintain reactor temperature. Other furnace components would fail rapidly under this changed condition.

Heat dissipation, the reverse of conductivity, resulted in 70 percent more output from an H_2-Cl_2 burner using a water cooled metal shell. Partial substitution of silicon carbide for the acid-proof refractory liner dissipated the heat at a faster rate and reduced the volume of HCl gases. This increased production in existing equipment.

Reactive conditions harmful to refractories often can be controlled by heat conductivity. High iron slags that become molten in boiler-furnace operations have a strong affinity for silicon carbide used in the walls. Positive air or water cooling reduces face temperature of the refractory sufficiently to congeal the slag and prevent attack. The same action occurs when the walls are metallic but more cooling is required.

Corrosion Resistance

High temperature conditions normally are combined with molten slags, metals and fluxes and corrosive fumes. Examples already cited show that special refractories resist many of these conditions.

Each corrosion problem requires separate study to consider not only the corrosive aspects but also processing conditions involved. A deviation of 200 F can change refractory recommendations that could mean success or failure for the installation.

Hot Compressive Strength

Hot strength is a measure of dimensional stability at various temperatures under load. Load comes from an external weight such as a furnace charge,

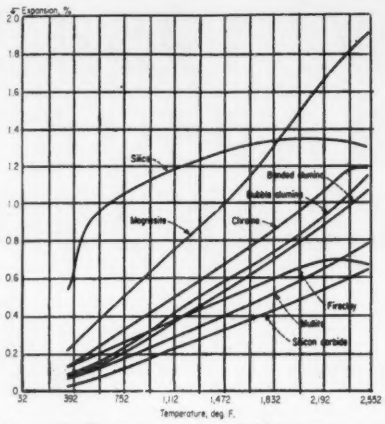

Figure 9—Thermal expansion of high temperature materials. A low, uniform expansion rate under heat as demonstrated by silicon carbide is a valuable asset in withstanding thermal shock.

from a superimposed load such as a furnace wall or from stresses within a structure itself such as expansion which occurs under heating conditions.

Hot strength of some materials of construction are given in Table 2. A demonstration of the importance of hot strength is shown in Figure 7.

For some applications, invaluable information about how materials carry loads is obtained from sustained tests that indicate performance over long periods and from short tests that give amount and uniformity of expansion to the point of maximum expansion. Low, uniform thermal expansion helps to avoid mechanical distortion of a structure.

Equally important is data on the temperature at which expansion ceases because softening usually follows. Shrinkage of only 5 percent is considered failure because this represents a 7/16-inch slump in a 9-inch long brick—sufficient to lose keying or wedging action.

Hot load bearing strength data are important considerations in large refractory structure such as linings of synthesis gas generators and domes in muriatic acid furnaces. Structural supports in hydrocarbon reformers are another example.

Abrasion Resistance

Results of Zeiss impingement tests on some non-metallics are given in Figure 8. The abrading material was fused aluminum oxide with a hardness rating of 9.0 on Moh's scale. The two basic types of abrasion are impingement by fine solids suspended in air, gas or liquid and direct abrasion by a mass of wet or dry solids moving across or at a surface.

Under these conditions, a materials' wear resistance is influenced by several variables: size, hardness, shape, weight and concentration of the abrasive particles. Velocity and direction of impact, temperature and corrosive nature of abrading material are other factors to be considered.

Bonded silicon carbide and some varieties of fused alumina are recommended for most high velocity impingement and some types of direct abrasion.

In some direct abrasion cases, impact is involved also. Heavy particles falling upon a surface can cause progressive bond fracture of clay-bonded materials. The refractory grog is mechanically lifted since the holding bond is destroyed. Similar action on monolithic linings results in chipping and gouging. Fused-cast refractories held together with interlocking crystals normally are the best solution to this kind of abrasion.

Extreme toughness and hardness of a material is an asset when abrasion and corrosion are combined. If hardness prevents a surface breakdown by abrasion, chemical attack is retarded. Should the bond be susceptible to reaction, the bondless fused-cast products are a logical selection.

Applications of abrasion resistant special refractories range from dust collectors, hoppers and gas scrubbers to chutes, transfer pipe lines and classifiers to rabble blades, acid slurry pump parts, spray nozzles and weir-wear blocks.

Heat-Shock Resistance

Rapid and repeated temperature changes can generate such high thermal gradients in refractory parts that severe spalling and cracking of the refractory are caused. Operation economics may preclude steps to lessen heat shock; therefore materials resistant to this condition must be investigated.

(Continued on Page 98)

TABLE 2—Comparative Data on Sustained and Short-Duration, High-Temperature Load Tests

Refractory Compositions	100 Hour 25 psi Load Test		1½ Hour 25 psi Load Test	
	Hold Temperature Degrees F	% Linear Cold Contraction	Hold Temperature Degrees F	% Linear Cold Contraction
Silicon-Nitride-Bonded Silicon Carbide	2732	0.0	2732	0.0
Silicate-Bonded Silicon Carbide	2732	0.58	2732 3146	Nil 0.78
Electric Furnace Mullite	2732	1.23	2732 3128	0.11 1.31
Bonded Fused Alumina	2462 2732	0.25 8.68	3130	8.86
Fused-Cast Beta Alumina	2732	0.0	2732	Nil
Converted Kyanite	....	....	3137	14.3
Chrome Magnesia	....	....	Crushed at 3002	Sheared
Magnesia	....	....	Crushed at 2552	Sheared
Silica	2732	0.13	Crushed sharply at 2948	Sheared
High alumina	2462	7.7	Squashed at 3011	25.46
Fireclay	2462	11.3	Squashed at 3002	25.0
Fireclay	2462	15.1	Squashed at 2993	26.6

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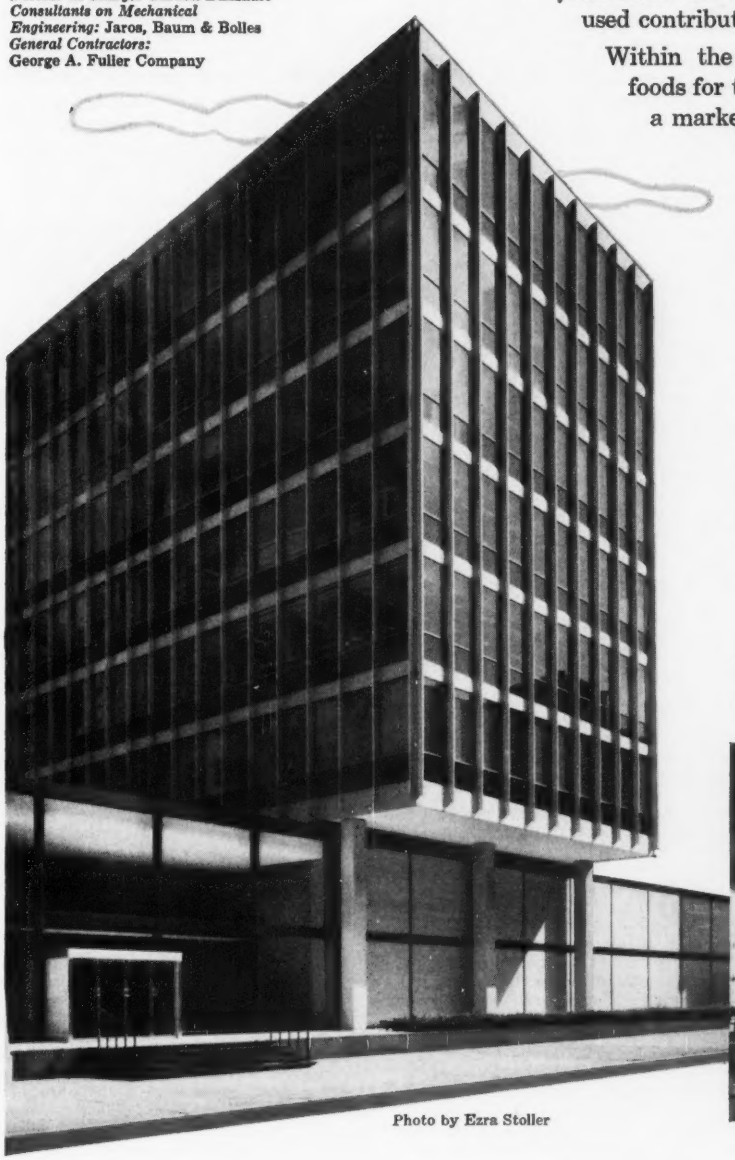

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Special Corrosion—

(Continued From Page 96)

Thermal conductivity, tensile strength, modulus of elasticity and thermal expansion have major influence on heat-shock resistance. Thermal expansion of high temperature materials is shown in Figure 9. Low thermal expansion helps prevent a differential expansion across cooled and uncooled portions of a structure. High thermal conductivity minimizes differences in thermal gradients. Hot strength is essential to meet the inevitable strains that develop. If the situation permits, the possibility of varying the magnitude of shock, furnace design, refractory shape and operating conditions merit attention.

Of the special refractories, clay and nitride-bonded silicon carbide are the most resistant to heat shock. Electric furnace mullite is a close second. In this specific property, these products are separated widely from the materials in the overall group.

Thermal-shock resistance combined with corrosion resistance is an important factor in nitride-bonded silicon carbide spray nozzles and clay-bonded silicon carbide gas reformer checkers and chemical waste incinerator baffles. Silicon carbide lined H_2-Cl_2 burners and electric furnace mullite lined hydrocarbon reformers resist spalling ordinarily caused by high thermal gradients through their walls.

Permeability

Fused-cast refractories with their structure of crystal-to-crystal junctions are almost impermeable to liquids and gases. Shapes, however, are limited to simple, heavy cross-section pieces. Self-bonded silicon carbide material is also an impermeable body having some of the unique properties noted and is available in a range of sizes.

Clay and nitride-bonded silicon carbide also have low permeability, limiting cold air passage to one or two cubic feet per hour. This factor is valuable in applications using externally heated tubes carrying hydrocarbon gases for cracking and reforming. A more permeable refractory tube wall would retard reactions and constitute potential explosion hazards.

These same materials can withstand a differential internal pressure of 5 to 10 psi without appreciable leakage. The possibility of making individual parts with butt joints, tongue and groove joints, lap joint or ball and socket joints is also an advantage.

Silicon carbide's low permeability has proved useful in pump parts subjected to molten metal and acid slurries, in floats, pouring ladles, dampers, spray nozzles, etc. Radiating domes used to heat gases or to break down solids also must be impermeable to prevent atmospheric contamination.

Adapting Special Refractories

Though comparable in some aspects to metals, special refractories differ markedly in other properties. Most of them rate high in modulus of rupture and hot compressive strength but possess relatively poor tensile strength. Nitride-bonded silicon carbide, for example, has a compressive strength of approximately 20,000 psi at 2400 F and at the same temperature a tensile strength of only 3500 psi. This carbide and others in the same general class of materials do not have a yield point. Although strong, they are brittle and will break before bending.

Frequently, this brittleness problem can be overcome or at least circumvented by redesigning the part or lining to take advantage of the high compression strength of the refractories. In other cases, the same effect is obtained successfully with holding devices.

If severe thermal shock exists, special refractories made in very large shapes and thick sections are undesirable. Often sectionalization is a solution. Generally, a small compact shape is less susceptible to heat shock than a large piece, especially one of irregular cross section. Removal and replacement of parts are facilitated also.

If there is impact, again a sectional lining can be the answer. Addition of a resilient packing or back-up material lessens impact force on the refractory part. Impact in this instance refers to falling bodies, blows struck with cleaning tools, etc.

Complexity of Shapes

Although fused-cast refractories can be made in 9-inch straight brick, the preferred and most economical shapes must be comparatively heavy cross sections and roughly rectangular. The large size and extreme density of these fused-cast materials lowers thermal shock resistance and confines usage largely to furnaces and equipment operated continuously without appreciable temperature fluctuations.

Clay-bonded special refractories are made in all brick sizes and in a great variety of patterns far more complex than normally furnished in the common types of refractories. Even so, they fail to match the intricate designs possible with metals.

Nitride-bonded silicon carbide formability is much closer to the metals. Exceptionally low volume change during drying and firing permits tolerances of ± 0.003 to ± 0.005-inch per inch when closer than normal tolerances are required. When cast, this material has a surface finish of approximately 250 micro inches; compacted and pressed parts attain a finish of 500 micro inches. Although usually unnecessary, surface grinding can and has been done. A finish of 32 micro inches has been accomplished with a diamond wheel. Self-bonded silicon carbide can be ground close to a mirror finish with a diamond wheel.

Some parts produced from silicon-nitride-bonded silicon carbide are formed with standard threads of good accuracy. Other techniques permit making separate pieces that on firing provide an assembly heretofore impossible to combine with ceramic joining methods. Figure 1 shows a representative group of complex shapes.

Because shape and size of a piece are essential to good performance, the following points should be remembered:

1. Avoid extremes in thickness and radical changes in section.
2. Very small parts are difficult and costly to manufacture.
3. A long, thin shape may tend to warp in firing.
4. Walls less than ¼-inch thick are fragile and cause production troubles.
5. Sections over 3 inches thick are undesirable because heat shock resistance is impaired.
6. Minimize length of part subjected to heat and cold. It might be better to use two shapes, one for the hot area and the other for the cold portion.

Summary

Corrosion resistance of special refractories makes them worthy of consideration for those areas of furnace and processing equipment when ordinary refractories, cermets and metals do not give satisfactory service. Study of their possibilities for this condition must include all their properties whether used individually or in combination. Frequently, it is possible to take steps that offset seemingly poor characteristics to permit usage of favorable ones.

DISCUSSIONS

Comments by William L. Wilson, Columbia-Southern Chemical Corp., Barberton, Ohio:

I was surprised to see silicon carbide used in hot chlorine. We generally feel that SiC is not very resistant to chlorine. Will you please comment on this?

Reply by Roy W. Brown:

This question points out the need for working closely with a reputable manufacturer who knows the limits of his product. Our work has indicated that ceramic bonded silicon carbide can be used to 600 C as it has been in the perchloroethylene unit. We know that it cannot be used above this temperature. Silicon carbide tends to react with hot chlorine gases to form silicon tetrachloride.

Comments by Roy E. Dial, Carborundum Co., Niagara Falls, N. Y.:

My work on selected, high grade, samples of dense silicon carbide shows that it will not withstand Cl_2 gas at 600 C. It does very well at 500 C. The effect of Cl_2 at temperatures between 500 and 600 C has not been determined.

Silicon nitride or silicon-nitride-bonded silicon carbide should be considered for Cl_2 at temperatures of 600 C and higher. I know from published data that Si_3N_4 is not attacked by Cl_2 at 800 to 900 C.

Reply by Roy W. Brown:

This is interesting additional information. Since the reaction seems to be with the silicon carbide crystal itself, it may well be that the ceramic bond of the kiln fired product may give it some additional protection over the dense silicon carbide made in induction furnaces without any bond.

Technical Topics Scheduled For November Publication

Accumulated Residues Attack Chemical Distilling Column, by H. Gilman

Can Corrosion Problems, by J. J. Daly, Jr.

Some Properties and Uses of Chlorinated Polyether, by C. S. Miller and J. B. Martin

Technical Topics Scheduled For Early Publication

Cathodic Protection of Southern Pacific's High-Pressure Products Pipe Line, by D. N. Miller

Corrosion Control Practices in the Wilmington Water Flood Operation, by C. H. Jones

Engineering Approach to a Chemical Plant Coating Program, by Newell D. Casdorph

Characteristic Properties of Polyurethane Protective Coatings, by E. R. Wells

CORRODION ABSTRACTS

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3. CHARACTERISTIC CORROSION PHENOMENA

3.2 Forms

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Hydrogen, Crack Initiation and Delayed Failure in Steel. H. H. Johnson, J. G. Morlet and A. R. Troiano, Trans. Met. Soc. AIME (Am. Inst. Met. & Mining Engrs.), **212,** 528-536 (1958) August.

Delayed failure in steel occurs by controlled initiation and growth of a crack. The incubation period for crack initiation was measured. Crack initiation and propagation are controlled by interaction between hydrogen concentration and triaxial stress state. The incubation period results from stress-induced diffusion of hydrogen to the point of crack initiation. (auth)—NSA. 17332

3.2.2, 6.4.4, 3.8.2

Mechanism of Stress-Corrosion Cracking in the AZ31B Magnesium Alloy. Research Paper No. 2919. Hugh L. Logan. J. Res. National Bureau Standards, **61,** No. 6, 503-508 (1958) Dec.

The mechanism of stress-corrosion cracking of annealed AZ31B magnesium alloy in an aqueous solution of 3.5 percent sodium chloride plus 2.0 percent potassium chromate was investigated. Cracking was predominantly an electrochemical process and was shown to be dependent on the rate of strain in the specimen following loading in tension. It is postulated that cracks develop if the protective film on the metal surface is ruptured (over narrow segments of the specimen) at a greater rate than it is repaired in the corroding medium. This would expose film-free metal that is anodic to the filmed metal.—ALL. 17480

3.2.2, 3.7.3, 7.2, 5.12

Ringworm Corrosion in Upset Tubing. J. P. Engle and C. E. Fox. Dow Chemical Co. World Oil, **147,** No. 2, 112-114, 116 (1958) August 1.

Ringworm corrosion is a special type of localized tubing corrosion associated with heat-affected areas or zones. These zones are produced during forging operations in forming upset ends on tubing. Such corrosion takes the form of a ring or band of pits, sometimes completely encircling tubing, a few inches from upset end. This circumferential pitting weakens tubing, and may result in expensive finishing jobs if weakened tubing should part. Best solution in prevention of ringworm corrosion is to subject tubing, after upsetting operations, to a normalizing heat treatment. Entire tube is heated above transition temperature, removed from furnace and allowed to cool. In this manner, entire tube is recrystallized, grain size is uniform throughout, pearlite phase is uniform throughout, and no sensitive zone of spheroidized pearlite remains. Chemical inhibitors that retard action of corrosive fluids on metal partially alleviate failure of sensitive parts. Photomicrographs.—INCO. 17239

3.2.2, 3.5.8, 3.7.4

Causes of Stress-Corrosion Cracking in Nonferrous Metals and Steels. (In German.) Ludwig Graf. Werkstoffe u. Korrosion, **9,** No. 11, 693-698 (1958) Nov.

Reasons for extremely localized attack on intercrystalline grain boundaries and the effect of tensile stress on stress-corrosion of steel, copper and copper-zinc and copper-gold. Tensile stress increases activity of solid solution in a state of plastic deformation, but does not cause localization of the intergranular attack. Latter effect appears in homogeneous solid solutions in the absence of stress; is traced to increased reactivity of obtuse-angled grain boundaries due to solid solution formation.—MR. 17409

3.4 Chemical Effects

3.4.3, 4.6.6

An Unusual Form of Corrosion Product. G. Butler and H. C. K. Ison. Nature, **182,** No. 4644, 1229-1230 (1958) Nov. 1.

During an investigation into influence of speed of flow on internal corrosion of mild steel tubes, an interesting and unusual form of corrosion product occurred. Tests were carried out with annealed ⅝ in bore pipes through which Metropolitan Water Board water was flowing at 56 C at a speed at 430 ft/min. Examination of sample lengths which had been on test for 4-10 weeks showed formation of a rough, very adherent scale on the surface with a number of "whiskers" growing perpendicular to tube surface and then curving and growing parallel to tube surface and along the direction of flow. The following tentative mechanism for formation of these whiskers is suggested. During early stages of corrosion an adherent carbonate scale is thrown down by the hard water, but this is not sufficiently complete or repairable, since the alkalinity rapidly falls, to stifle corrosion completely and corrosion takes place at local anodic points. In stagnant solution localized corrosion commonly leads to formation of a hemispherical dome of corrosion product over a pit. It appears that under the influence of rapidly flowing water the symmetry of the form of corrosion product is disturbed, leading to formation of a hollow cylinder bent through a right angle in direction of flow. Ferrous ions, formed at anodic area at root of whisker, diffuse along tube and growth of whisker proceeds at the tip, where these ions are oxidized and precipitated as iron oxide on coming into contact with the aerated water. Rate of ionic diffusion may be increased by suction induced by rapidly flowing water at the tip. Photomicrograph.—INCO. 17160

3.4.8

Corrosion of Steel by Hydrogen Sulfide. Progress Report for Period September 15-December 11, 1952. J. C. Rowell. E. I. du Pont de Nemours & Co. U. S. Atomic Energy Commission Pubn., ESP-52-351, Jan. 8, 1953 (Declassified March 20, 1957), 17 pp. Available from Office of Technical Services, Washington, D. C.

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The role of the iron sulfide scale in hydrogen sulfide corrosion of steel is investigated. The structure, characteristics and mechanism of formation of the iron sulfide are given.—NSA. 13626

3.4.8, 5.8.1

Influence of Bicarbonate Ion on Inhibition of Corrosion by Sodium Silicate in a Zinc-Iron System. Henry L. Shuldener and Leo Lehrman. J. Am. Water Works Assoc., **49**, 1432-1440 (1957), Nov.

Effect of zinc, silicon dioxide, pH and temperature on corrosion of rotating galvanized pipe. 21 references.—BTR. 16340

3.4.9, 6.2.5, 3.2.2

Effects of Superheated Water Vapor on the Austenite Steel Resistance. (In Russian.) E. A. Davidovskaya and L. P. Kestel. Central Research Inst. of Machine Building and Metalworking. Metalloved. i Obrabotka Metallov. No. 8, 29-33 (1958) Aug.

It is shown that pure superheated vapor does not affect the stabilized resistance of austenite at 600 C; however, the presence of various salts may affect the durability of steel, especially at high tension.—NSA. 17248

3.5 Physical and Mechanical Effects

3.5.4, 6.3.10, 6.3.6, 3.5.9

The Effect of Neutron Irradiation on the Mechanical Properties of Copper and Nickel. M. J. Makin. J. Inst. Metals, **86**, Pt. 10, 449-455 (1958) June.

Effect of neutron irradiation at 100 C with 5×10^{19} neutrons/cm^2 on the mechanical properties of annealed polycrystalline copper and nickel was studied at a variety of temperatures. Yield stress of both metals is increased by irradiation and the increase is markedly temperature-dependent, being very much greater at low temperatures than at high. Temperature variation in yield stress after irradiation is a reversible phenomenon and is not due to an annealing effect at the higher temperatures. Rate of work-hardening is greatly reduced by irradiation, especially at low strains. Ultimate tensile strength is increased and elongation to fracture is reduced by irradiation. Results are discussed in terms of possible mechanism of irradiation-hardening. Graphs.—INCO. 16908

3.5.4, 3.4.3

Van de Graaff Study: Deposition of Corrosion Products Under Irradiation. G. P. Simon, B. G. Schultz and Y. Solomon. Bettis Technical Review, **1**, No. 3, 39-52 (1957) Aug. U. S. Atomic Energy Comm., WAPD-BT-3.

Ionizing radiation definitely results in the deposition of "freshly-produced" corrosion products. Form of corrosion products most efficiently deposited from loop water is colloidal or soluble. Since both particulate corrosion products and ferrous ion are not efficiently deposited, mechanism of partial hydrolysis and oxidation to form a colloid which reacts with radicals, produced by ionizing radiation, to produce deposition is supported. Amount of deposition under irradiation is a function of amount of colloidal or soluble corrosion products available. Zircaloy-2 was specimen used in test. Diagrams, graphs.—INCO. 17045

3.5.4, 3.8.4, 6.3.19, 6.2.2

Corrosion Studies. Pt. XI. The Influence of Light on the Atmospheric Corrosion of Zinc and Iron in a Pure Atmosphere. (In German.) E. Beranek, K. Barton, K. Smrcek and I. Sekerka Coll. Czechoslov. Chem. Commun., **22**, No. 2, 368-378 (1957).

The differences found in the corrosion of zinc by air saturated with water vapor at 20, 30 and 40 C in the dark and when illuminated with ultra-violet light, and the apparent dependence of the potential of a zinc electrode on the presence or absence of illumination, suggests a photochemical process as responsible for the corrosion. The results of further experiments show that the enhanced corrosion obtained by illumination is still further enhanced by the presence of hydrogen peroxide. These results are discussed in terms of the decomposition of the oxide layer, thus $ZnO \xrightarrow{2h\nu} Zn^{2+} + 2e + \frac{1}{2}O_2$; $Fe_2O_3 \xrightarrow{2h\nu} 2(Fe^{3+}O^{2-}) + 2e + \frac{1}{2}O_2$. 18 references.—MA. 15747

3.5.8, 2.3.7, 6.3.10, 3.5.9

Fatigue of Nickel-Chromium Alloys at Elevated Temperatures. A. W. Franklin and E. D. Ward. Mond. Rev. Met., **55**, No. 10, 927-938 (1958) October.

Presents results of fatigue tests on Nimonics 80A, 90, 95 and 100, carried out at temperatures ranging 700-870 C. Fatigue strength varies in same direction as creep strength. Comparison is made of metallographic characteristics of fractures in creep and in fatigue. Notches have no notable effect on fatigue test results. Numerous tables, graphs, photomicrographs.—INCO. 17265

3.5.4, 3.4.8, 4.4.6, 6.2.5

Radiation Stability of Carbon Tetrachloride. R. E. Olson and K. L. Adler.

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General Electric Co., Hanford Atomic Products Operation. U. S. Atomic Energy Commission Pubn., HW-32405 (Del.), July 12, 1954 (Declassified with Deletions Feb. 26, 1957), 14 pp. Available from Office of Technical Services, Washington, D. C.

The decomposition of carbon tetrachloride containing TBP exposed to γ radiation is roughly proportional to the total radiation exposure. Excessive corrosion rates due to high Cl^- ion concentrations may be expected on stainless steel equipment exposed to solutions which have been in contact with carbon tetrachloride at high radiation intensities. Corrosion may be particularly severe in equipment operated at high temperatures and in the presence of hydrogen ion. (auth)—NSA. 17031

3.5.4, 4.7, 6.2.5

Corrosion of "Croloy" Steel by Liquid Bismuth Under Cyclotron Irradiation. N. S. Rasor and A. A. Epp. North American Aviation Inc., U. S. Atomic Energy Commission Pubn., NAA-SR-MTA-24 (Del.), Declassified with Deletions April 16, 1957, 16 pp. Available from Office of Technical Services, Washington, D. C.

The proton beam from the Crocker 60 in. cyclotron incident on a liquid bismuth-Croloy steel interface has been used to determine the effect of intense radiation on the rate of corrosion. The results indicate that under the specified atmosphere and temperature conditions there is no large effect on the rate of corrosion of Croloy steel by liquid bismuth due to the presence of the radiation. A possible increase in the rate of wetting by bismuth due to the radiation is indicated. Factors affecting the results observed are reviewed and improvements in technique for future experiments suggested. (auth)—NSA. 17092

3.5.8, 3.5.9

Thermal Stresses in Design. Part 4—Causes of Fatigue in Ductile Materials. S. S. Manson. Lewis Flight Propulsion Lab. Machine Design, **30,** No. 17, 110-113 (1958) August 21.

Discusses effects of temperature variations, hold time, sign of strain and degree of constraint in analysis of thermal-stress fatigue. Data are presented graphically for Inconel 550, S-816 and Type 347.—INCO. 17482

3.5.8, 5.12

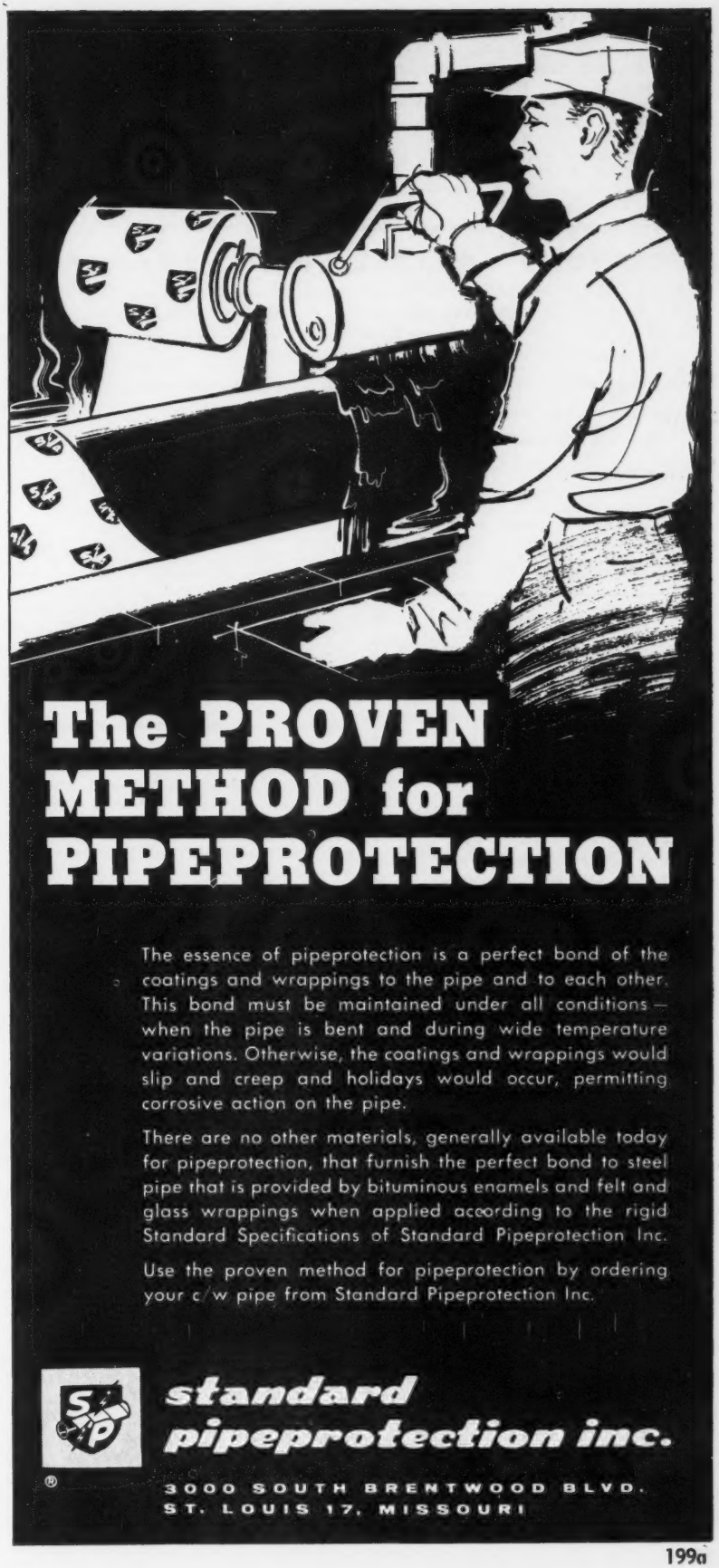
Stress-Corrosion. (In German.) Ludwig Graf. Draht, **9,** 383-389 (1958) Oct.

Stress-corrosion sensitivity of supersaturated alloys with precipitation tendency can be eliminated by heat treatment, provided the alloy is not corrosive sensitive in the homogeneous state of saturation. With unsupersaturated homogeneous alloys, stress-corrosion was observed only when the alloying components of the solid solution were considerably more negative than the base metal. 19 reference.—RML. 17398

3.5.8, 3.5.9

Thermal Stress in Design. Part 5—Interpretation of Fatigue Data for Ductile Materials. S. S. Manson. Lewis Flight Propulsion Lab. Machine Design, **30,** No. 18, 126-133 (1958) September 4.

Reviews mechanical properties under cycling, prior straining, distinction between thermal and mechanical fatigue and design applications. Most important single variable in thermal-stress cycling is maximum temperature of cycle. In-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creasing maximum temperature, for given temperature range, causes much greater reduction in cycles to failure than increasing temperature range by same amount and maintaining same maximum temperature. Time at which maximum temperature is maintained has a lesser effect. Thermal-stress fatigue is quite analogous to mechanical fatigue, and preliminary indications are that both are governed by similar mathematical relations. Because of continuously changing temperature during thermal-stress fatigue test, and because of strain concentration and localization effects, it is difficult to predict life in thermal fatigue from mechanical fatigue at constant temperature. Effect of prior thermal-cycling on specimens subsequently evaluated in stress-rupture depends greatly on material and number of prior cycles. Effect is generally detrimental. For precipitation-hardened alloy such as S-816, in which further aging produces beneficial effects, small number of thermal-stress cycles increases resistance to stress rupture. Each of materials studied to date—Type 347, S-816, Inconel and Inconel 550—displays characteristics considerably different from others in thermal-stress fatigue. Graphs show room-temperature tensile ductility of Type 347 as affected by prior thermal cycling; various relations between thermal-stress, hardness and stress-rupture life and fatigue life for S-816 and Inconel 550.—INCO. 17483

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3.7 Metallurgical Effects

3.7.3

Further Evaluation of Nickel-Chromium-Boren and Gold-18 Nickel Brazing Alloys. W. H. Chang. Gen. Elec. Co. Welding J., **37,** No. 12, 535s-542s (1958) Dec.

Tensile and impact properties of AISI 347 and AISI 410 butt joints brazed with nickel-chromium-boron alloy and with gold-18 nickel alloy are evaluated. Data are presented to show changes of these properties as affected by temperature and/or joint clearance between room temperature and 1200 F. These changes are discussed from consideration of characteristics of brazing alloys as well as base metals. In addition, some data are obtained on 1500 F oxidation resistance of brazed joints. Tables, graphs, photomicrographs.—INCO. 17219

3.7.3

Automatic Welding of Aluminium with a Split Electrode. (In Russian.) F. S. Bugry. Svarochnoe Proizvodstvo, No. 5, 27-29 (1958).

Welding of circular butt joints with self-propelled welding heads with split electrodes considerably simplifies and speeds up the welding of large cylindrical vessels used in the chemical industry and affords better corrosion-resistance of the weld metal.—MA. 17152

3.7.3

High-Temperature Corrosion-Resistant Brazing. J. V. Long and G. D. Cremer. Solar Aircraft Co. Astronautics, **3,** No. 8, 28-29, 86-87 (1958) August.

Reviews developments in high-temperature corrosion-resistant brazing, process introduced less than 8 years ago. Advantages include: ability to join complex, light-weight components; great flexibility in joining light and heavy sections; high reliability of parts because of high joint strength and fatigue and impact resistance; high strength at high temperatures; more freedom from localized stresses; better distribution of thermal stresses; and higher production rates with reduced costs. Problem of compression disappears when thin sheet metal is combined with honeycomb core and brazed into sandwich structure. No other fabricated structure is as efficient on strength-to-weight basis. Basic sandwich can be modified to provide improved insulation, internal pressurization and fluid flow. Refractory fillers in core increase thermal resistance. Photos show stainless steel honeycomb sandwich and Inconel-faced honeycomb sandwich door for electric furnace.—INCO. 17479

3.7.3, 5.12

Effects of Preheating, Thermal Stress Relief and Electrode Type on Notch Toughness. L. J. McGeady. Lafayette

College. Welding J., **37,** No. 12, 543s-553s (1958) Dec.

Bend test of welded flat-plate specimens, 1¼ in. thick and 2 ft. square, were made using heat of A-201 Grade A steel. Weld was full butt joint down length of specimen, long enough for yield-point residual stresses to be set up in metal. Plates were welded without preheat and with preheats of 250 and 400 F. E6010 and E6016 electrode types were used. Several series of plates welded without preheat were thermally stress relieved. Supplementary tests were made on Charpy-V test bars. Heat of steel to A-212 Grade B was tested in many of the above tests. Tables, graphs.—INCO. 17473

3.7.3, 6.2.4

Study on Weldability of Low Alloy Steels. H. Suzuki, H. Tamura, T. Oda and K. Tajima. Weld. Rev., **5,** No. 1, 15-26 (1957) Jan.

Weldability and choice of electrodes for two kinds of high-strength low-alloy steels, namely nickel-molybdenum steel tempered after quenching and nickel-chromium-molybdenum steel tempered after normalizing. The results of various tests such as V-Charpy impact, notched bead bend (Kinzel), slit-type cracking (Tekken), frame-cut bend and welded joint tests have shown that the welding quality of these steels can be sufficiently improved either by tempering at about 630 C after quenching or normalizing, or by pre-heating at above 200 C prior to welding.—INCO. 17138

3.7.3, 6.2.5

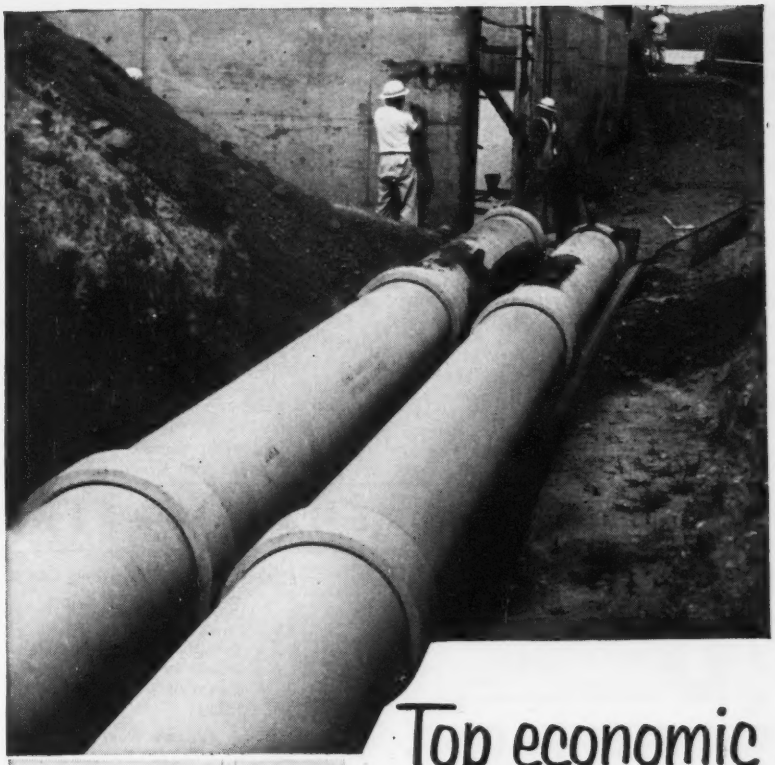
An Investigation of Lap Seam Welds in 17-7 PH Stainless Steel. E. J. Funk and A. C. Willis. Temco Aircraft Corp. Paper before Am. Welding Soc., Annual Spring Mtg., St. Louis, April 14-18, 1958. Welding J., **37,** No. 9, 897-905 (1958) Sept.

Describes method for measuring secondary current in seam welder with direct energy 3-phase primary, and for calibrating measuring equipment; and presents data on seam welding of 17-7 PH sheet used in TH-1050 condition. These data include effect of certain welding variables (welding speed, heat cycling and surface preparation), and nugget width and penetration. Discussion covers defects found and reduction of joint strength at elevated temperatures as determined by tension-shear testing. Tables, graphs, photomicrographs.—INCO. 17255

3.7.4, 6.2.2

Effect of Graphite Shape and Matrix Structure on Hardness of Ductile Cast Iron. (In Japanese.) R. Kusakawa. J. Japan Soc. Mech. Engrs., **61,** No. 477, 1180-1186 (1958) October.

Reasons why hardness of ductile iron is superior to flaky graphite cast irons are explained. Due to the spheroidization of graphite, the surface area of graphite becomes minimum, so that the strain at weak interface between graphite and matrix is minimized, thus the hardness of the iron approaches to that of matrix. The hardness of matrix in pearlitic ductile iron may increase due to the fact that pearlitic structure is easily transformed into fine sorbitic structure by super-cooling in the casting practice. Iron tends to transform into white cast iron by magnesium treatment thereby the hardness is remarkably increased by precipitation of cementite. The hardenability of ductile iron is proved excellent as a result of Jominy test. Hardness is increased because the structure is consisting of both



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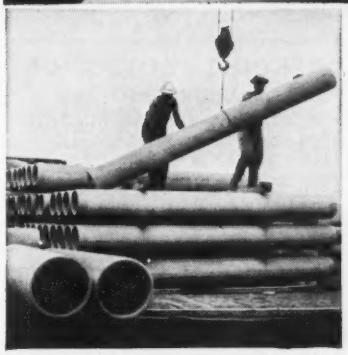

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heat treated matrix and spheroidal graphites. Addition of magnesium with nickel, manganese, etc. may move the isothermal transformation curve to the right, and when the curve moves further to the right the hardness would increase.—INCO. 17441

3.7.4, 6.3.3, 2.3.9

Cleavage Fracture in Cast Chromium of High Purity. J. F. McNeil and H. R. Limb. J. Inst. Metals, **87**, Pt. 3, 79-87 (1958) Nov.

Surfaces produced by brittle fracture of cast, coarse-grained, high-purity polycrystalline chromium were examined in detail by means of fractography, goniometry and X-ray diffraction. Although many features of the fracture of chromium by cleavage are similar to those reported for other metals, there are some features present which have previously been noted only in the brittle fracture of vitreous materials. Various types of markings on the cleavage surfaces are explained in terms of the fracture process and where possible related to the crystal lattice. Four types of cleaved grain were distinguished on this basis. Cleavage takes place mainly on (001), to a minor extent on (112), and exceptionally, on or close to (111) planes; simultaneous cleavage on a number of planes in (110) or (100) zones also occurs in isolated grains. Under certain conditions cleavage may deviate by as much as 10° from (001) plane, to produce curved facets. Photomicrographs, tables, 30 references.—INCO. 17475

3.7.3, 7.4.4, 6.2.5

Seven Methods for Joining Stainless Steels. P. Bowman. Whirlpool Corp. Ind. and Welding, **31**, No. 8, 42-43, 63 (1958) August.

Description of different methods of welding used to manufacture bulk milk coolers. Coolers are made in 155 to 735 gallon capacities and are of low carbon type 302 stainless; 14 gauge for inside liners, 16 gauge for exterior shells. Photos.—INCO. 17164

3.7.4

On Source of Dislocations in the Plastic Deformation of Crystals. (In German.) D. Kuhlmann-Wilsdorf, R. Maddin and H. Kimura. Z. Metallkunde, **49**, No. 11, 584-589 (1958) Nov.

Arguments against theory that majority of dislocations, formed during slip, are released from random 3-dimensional dislocation networks formed during crystallization, are presented. It is proposed that so-called R-dislocations are sources of dislocations in plastic deformation. These dislocations form boundary of single layers of condensed thermal vacancies, parallel to most densely packed lattice planes. Since condensation of thermal vacancies constitutes an evaporation (a negative crystallization), it is supposed that R-dislocations consist of seg-

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ments parallel to most densely spaced lattice directions. Recent observations are discussed wherein predicted R-dislocations were found in thin foils of quenched aluminum and nickel, examined directly in electron microscope.—INCO. 17447

3.7.4, 6.3.10, 2.3.9, 3.7.2

Crystallographic Structure and Orientation of the λ Phase in Four Commer- W. C. Bigelow. Univ. Mich. U. S. Wright Air Development Center, Technical Note No. 57-247, July, 1957, 12 pp. Available from Office of Technical Services, Washington, D. C. (Order PB 131518).

Selected area electron diffraction patterns were obtained from matrix precipitate particles siolated by extraction-replica technique from aged specimens of Inconel-X, Waspalloy, M-252 and Udimet, which contain titanium and aluminum as hardening agents. Matrix particles were conclusively identified as λ′ phase. Ordered, or superlattice structure reported for this phase in simpler alloy systems occurs also in these complex alloys. Particles develop with high degree of preferred crystallographic orientation relative to matrix lattice. Variations in size, shape and distribution of particles in specimens of the 4 alloys aged 100 hours at 1400 F are also evident, and appear to be related to titanium and aluminum content of alloys. Table lists interplanar spacings and relative intensities of electron diffraction patterns from λ′ phase. Electron micrographs, diffraction patterns.—INCO. 16991

3.8 Miscellaneous Principles

3.8.4

Ionic Conductivity of Anodic Films at High Field-Strengths: Transient Behavior. D. A. Vermilyea. J. Electrochem. Soc., **104,** No. 7, 427-433 (1957).

The changes in ionic conductivity in tantalum pentoxide films following rapid changes in applied voltage, or following a heat-treatment show that activation energy and activation distance for ionic motion are functions of electrical field temperature. It is suggested that changes in the applied field change the local configuration and thus the mobility of the ions. Similar short term behavior is shown in aluminum oxide and niobium pentoxide films. 12 references.—MA. 17084

3.8.4

Oxidation of Metals: Determination of Activation Energies. Per Kofstad. Central Inst. for Industrial Research, Blindern, Oslo. Acta Chem. Scand., **12,** No. 4, 701-707 (1958).

A new method is proposed for the determination of energies of activation of oxidation of metal and alloys. Metals are oxidized under conditions of linearly increasing temperature, and the activation energy may be determined from one single oxidation run. In addition the method also gives the approximate temperature regions in which different rate laws of oxidation are found. The described method has been applied to high-temperature oxidation of nickel, zirconium, copper and titanium. (auth)—NSA. 17429

3.8.4, 3.5.8

Determination of the Course of Poisson's Ratio During Elastic and Slight Plastic Strain. (In German.) W. Köster and J. Scherb. Z. Metallkunde, **49,** No. 10, 501-507 (1958) October.

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Poisson's ratio of rhodium and iridium was found to be 0.26. Stress-strain curve in elastic region consists of 2 straight lines with different slope. Only slight change of Poisson's ratio was observed in this elastic region. Values were determined for platinum, palladium, silver, copper, gold and aluminum during and after plastic deformation. At beginning of plastic flow Poisson's ratio may reach values up to 0.5. Small plastic deformation (~ 1 per cent) has no effect on Poisson's ratio. For silver, Poisson's ratio decreases at higher deformations. Behavior of Poisson's ratio at low stresses is discussed. Table, graphs.—INCO. 17421

3.8.4, 3.8.3, 4.3.2, 6.3.21

The Reaction of Germanium with Nitric Acid. Pt. I. The Dissolution Reaction. Pt. II. Passivity of Germanium. Mary C. Cretella and Harry C. Gatos. J. Electrochem. Soc., **105,** No. 9, 487-492, 492-496 (1958).

Pt. I. The dissolution rate of single-crystal germanium in nitric acid increased with nitric acid concentration up to 6N and decreased with stirring. As the rate is a function of concentration of undissociated nitric acid and HNO_2, and as HNO_2 is a reaction product, the reaction is autocatalytic. The dissolution potential of both p- and n-type germanium becomes more noble with increasing nitric acid concentration, and the potential of n-type germanium is more noble than p-type. Hydrofluoric acid does not affect the dissolution rate unless the concentration is high enough ($> 6N$) to decrease the dissociation of nitric acid.

Pt. II. Above 6N-nitric acid the solution rate of germanium decreases, and for a given concentration the rate decreases with time, tending to zero. The higher the nitric acid concentration the sooner germanium becomes passive. The passivity is associated with an oxide film, which is soluble in hydrofluoric acid. Thus hydrofluoric acid prevents passivity. 25 references.—MA. 17197

3.8.4, 4.3.3, 3.5.9, 6.3.10

Oxidation of Nickel in Sulfur Dioxide at Elevated Temperatures. (In Russian.) V. V. Ipat'ev and D. V. Zheltukhin. Kirov Leningrad Academy of Tech. and Forestry. Metalloved. i Obrabotka Metallov., No. 12, 42-45 (1958) Dec. Translation available from: Henry Brutcher, Technical Translations, P. O. Box 157, Altadena, Calif.

The oxidation of nickel in sulfur dioxide at 600 to 800 C follows a parabolic law, scale formation is at the maximum at 800 C, and the oxidation rate is much faster than in air. The scale formed has two phases, nickel monoxide and nickel monosulfide. The scheme of oxidation is $3\ Ni + SO_2 = 2\ NiO + NiS$.—NSA. 17384

3.8.4, 6.2.2, 3.4.9, 3.5.9

Reaction Between Iron and Water in the Absence of Oxygen. J. Electrochem. Soc., **105,** 322-324 (1958) June.

Reaction at 25, 60 and 300 C. At room temperature the primary product appears to be ferrous hydroxide. Formation of ferrous ferric oxide occurs readily in the iron-water system at both 60 and 300 C. It is possible that the ferrous ferric oxide is produced via formation and subsequent decomposition of ferrous hydroxide. 13 references.—MR. 17390

3.8.4, 6.2.5

Surface Reactions on Stainless Steels. (In French.) T. N. Rhodin. U. S. Atomic Energy Commission Translation No. 3287, for Oak Ridge National Lab. J. Chim. Phys., **54,** 72-89 (1957).

The characteristic chemical reactions which are produced on the surface of an alloy reflect a dynamic equilibrium between the influence of factors such as the composition of the gaseous phase, the surface structure, the properties of the film and the composition of the alloy. Three gas-metal reactions on the surface of stainless steels; adsorption, oxidation and decarbonization are considered. The experimental techniques used were microgravimetry under vacuum, electron microscopy and the micro-analysis of surface residues.—NSA. 17055

3.8.4, 6.4.4

On the Mechanism of the Anodic Dissolution of Magnesium. B. N. Kabanov and D. V. Kokoulina. Doklady Akad. Nauk S.S.S.R., **120,** 558-561 (1958). Translation by Morris D. Friedman, Lincoln Lab., Massachusetts Inst. of Technology, U. S. Atomic Energy Translation NP-tr-153.

The mechanism of the rapid stage in the presence of a slower one for the process of anodic oxidation of a metal has not been explained successfully because of the experimental and theoretical difficulties. This appears to be possible at the present time in the case of metals which form a monovalent ion, unstable in solution, as the transition state during anodic dissolution. The process of magnesium dissolution in acid solutions is given. The oxidation of metallic magnesium up to the monovalent stage is the slowest of the electrochemical stages of this process since two parallel stages proceed after it, of which the diffusion into the solution is almost always rapid because of the instability of the monovalent ions. The electrochemical oxidation of metallic magnesium is retarded very intensely when it adsorbs oxygen, i.e., it is passivated. (auth)—NSA. 17361

3.8.4, 6.3.20

Studies on the Oxidation of Zirconium in Air. (In Japanese.) Tatsuo Maekawa, Takuya Hata, and Yoshihiko Tachihara. J. Japan Inst. Metals, (Nippon Kinzoku Gakkai-Si), **22,** No. 3, 150-153 (1958).

The reaction kinetics of reactor-grade zirconium at atmospheric pressure were studied at 650-900 C. The rate laws were deduced and the activation energy calculated (48.4 kcal/mole). The absorption of nitrogen during the oxidation was also studied and an activation energy of 82.4 kcal/mole obtained. The rate-controlling factor in the oxidation of zirconium is the diffusion of oxygen ions in the oxide film. 14 references.—MA. 17492

3.8.4, 6.3.20

Anodic Oxide Film Formation of Zirconium Kinetics With and Without Concurrent Oxygen Evolution. George B. Adams, Jr., Tien-Shuey Lee, Samuel M. Draganov and Pierre Van Rysselberghe (Univ. of Oregon, Eugene). J. Electrochem. Soc., **105,** 660-665 (1958) Nov.

Comparative studies were made with zirconium to determine whether differential formation fields measured below oxygen evolution potentials agreed with those measured at high voltages. It was found that the low-voltage field is some fifteen to thirty percent higher than the corresponding field obtained at high voltages. However, the field lowering of the activation energy for the anodic oxidation is the same whether derived from

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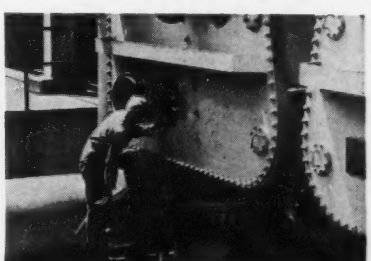

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high or low-voltage measurements. This is also true of the activation energy. These results confirm the view that the metal is anodically oxidized to the corresponding oxide at unit current efficiency below the reversible oxygen evolution potential. Ionic current efficiencies are reported for zirconium at several current densities in the high-voltage range and for the low-voltage localized oxygen evolution process. (auth)—NSA. 17157

3.8.4, 6.3.20, 3.7.4

Formation of the Delta-Phase by Oxidation of α — Titanium. I. Koncz and M. Koncz-Déri. Polytechnical Univ., Budapest. Periodica Polytechnica Chem. Eng., **1,** No. 1, 67-87 (1957).

The oxidation of iodide titanium was studied at a temperature range of 570 to 820 C, and a pressure range of 2 to 5 mm mercury in pure oxygen, for periods up to 210 minutes. Dense, adherent oxide layers show rectifying properties, the presence of delta-phase titanium in these layers was stated and verified by x-ray analysis with tetragonal cell dimensions: $a = 3.20$ A, $c = 5.12$ A, $c/a = 1.600$. The diffusion coefficient of oxygen in alpha-titanium is determined at 840 C as 5×10^{-11} cm^2 sec^{-1}. A possible explanation for the mechanism of the oxidation process is given. (auth)—NSA. 17007

5. PREVENTIVE MEASURES

5.2 Cathodic Protection

5.2.1, 7.3, 8.9.5, 3.5.3

Propeller Corrosion and Cavitation Erosion—Its Prevention by Cathodic Protection. M. G. Duff. Corrosion Technology, **5,** No. 8, 250-253 (1958) Aug.

Practical experiences with cathodic protection of ships' propellers on small vessels are described. Propellers and accessories are discussed for: bronze propellers on steel vessels, cast iron propellers on steel vessels and bronze propellers on wood vessels. The author maintains that cavitation and erosion are not the main causes of propeller damage, but are secondary in importance to corrosion. If application of cathodic protection to propeller resulted in cessation of all breakdown, then any breakdown which previously occurred must be attributed to corrosion mechanisms, and could not be primarily caused by physical means. This position has been reasonably established, and findings are generally applicable to bronze propellers of any size, and may be applicable to cast iron propellers. Recent protective schemes fitted to group of fishing vessels, to R.A.F. launches, and to 48-ton motor yacht, are cited. Theory of propeller breakdown is outlined. Mention is made of cavitation hammering effects on Monel propeller.—INCO. 17273

5.2.2, 3.6.5, 8.9.5

Investigation of Cathodic Protection of Ships' Hulls. (In German.) Romuald Juchniewicz. Werkstoffe u. Korrosion, **9,** No. 6, 360-368 (1958) June.

Electrode-potential measurements are described on ships' hulls using specially designed silver/silver chloride and copper/cuppric sulfate electrodes. Variation of potential around the hull and with ship speed is given, as well as the effect of protection by anti-fouling paints. Tests are also described on the corrosion of a model ship. The protection of ships' screws and tanks by means of zinc and magnesium electrodes is dealt with, as well as the use of a new cathodic control-system using a 5-6 mm soft aluminum wire trailed behind the ship as soluble anode. 36 references.—MA. 17393

5.2.1, 8.2.1, 8.2.2, 8.9.5

Danish Experience with Cathodic Protection. (In Danish.) H. Arup. Ingeniren, **67,** No. 8, 270-274 (1958) April 15; Sci. Absts. Section B, **61,** No. 727, 321 (1958) July.

The basic principles of cathodic protection are explained. Earlier experience with the Copenhagen district heating system showed that complete protection from corrosion due to tramway leakage currents could be obtained using superimposed currents and the tram rails as anodes over 62 km. of heating system with only 600 W. power consumption. Protection of ships is discussed. The Danish vessel Blenda has its underwater bolts protected with magnesium anodes. The protection achieved after one year's service is illustrated. Superimposed currents have not been used so far on Danish seagoing vessels but six laid-up ships were protected using old railway lines as anodes with only 20 amp., 1.5 V. from a rectifier at a cost of only 3 ore/-ship/day. Power station condensers and coolers can also be protected using either magnesium or zinc anodes. Examples of cathodic protection of steel underwater structures of quaysides and piers in Denmark are also described.—ALL. 17321

5.2.2, 8.9.5

Cathodic Protection Under Present Freight Conditions. Corrosion Technology, **5,** No. 8, 254-256 (1958) Aug.

Reviews economic advantages of using cathodic protection on tankers, and describes system being fitted by Seaguard Ltd. to black oil tankers ranging from 3,400 tons to 42,000 tons. Success of system is due to inherent simplicity of design and installation. Life and current output of anode is directly proportional to volume and surface area of anode, with reference to electrolyte in which action is taking place. Flat surface with small volume has comparatively high current output and short life; sphere has smallest surface area to volume ratio possible. Seaguard combined these 2 shapes in form of spherical collared magnesium anode. Installation in cargo tanks, deep tank protection, and hull protection are discussed.—INCO. 17231

5.2.3

Lead Alloy Anode for Cathodic Protection. J. H. Morgan. Corrosion Technology, **5,** 347-352 (1958) Nov.

Method of operation of a corrosion product impressed current anode and the formation of a conducting oxide film on a P6 alloy. Two types of impressed current anode can be used in sea water—consumable anodes from which metal is dissolved by the anodic reaction and permanent anodes which are not affected by the anodic reaction.—MR. 17496

5.2.2, 8.9.5

Cathodic Protection for Ships in Layup. G. W. Kurr and R. D. Taylor. Am. Smelting and Refining Co. World Ports and Mariner, 20, No. 9, 49-52 (1958) June.

Cathodic protection of underwater hull areas and ballasted tanks of laid up ships for short or prolonged periods effective protection against corrosion, is low in cost, simple to install and check, requires little or no maintenance, and the system can be removed in a matter

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5.2.3, 4.5.3, 1.2.2

Cathodic Protection of Cables by Tapping (the Current): Economy of the System. G. Fauvel. Corrosion et Anticorrosion, **5**, No. 11, 331-338 (1957).

The two main factors are 1, the length of cable protected by each tapping site, and $x\phi$, the dimensions of the anodes. An example is given of the calculation of 1 and the cost of protection/km., on the basis of an arbitrary value for the voltage drop in the coating at the tapping point, which permits assessment of the economic delivery. The $x\phi$ values are calculable as a function of the predicted protective-current strength and the soil resistivity. The characteristics of the rectifiers employed are determined in terms of 1, the diameter of the cable and the average resistance of the insulation of 1. —MA. 16193

5.2.4

Calculation of Cathodic Protection, Taking into Account the Interaction of the Fields in the Electrolyte. (In Russian.) V. V. Gerasimov. J. Applied Chem. (Zhur. Prikladnoi Khim.), **31**, No. 6, 946-948 (1958).

A method of calculating the distribution of potential and current density over a cathodically-protected metal system is described. Calculated and experimental data are compared for iron protected by magnesium cathodes.—MA. 17408

5.3 Metallic Coatings

5.3.2

Zinc and Cadmium Coatings as Protection Against Corrosion. (In German.) J. Elze and W. Wiederholt. Metallwaren-Ind. u. Galvanotech., **49**, 419-432 (1958) Oct.

Zinc is more durable in industrial and city atmospheres while cadmium is superior in conditions where condensation develops. Both are well suited for coating steel. 5 references.—MR. 17274

5.3.2, 2.2.7, 6.2.3

The Behavior of Zinc Coatings in Marine Conditions. Corrosion Prevention and Control, **5**, No. 9, 45-48, 74 (1958) Sept.

Coatings of aluminum, cadmium, lead, tin, zinc, 88-12 cadmium-tin and 88-12 lead-tin alloy were applied to mild steel specimens containing 0.2 per cent carbon. After two years totally submerged in sea water all the zinc coatings were found to be intact; aluminum coatings applied by molten metal or wire pistols were almost intact; cadmium behaved fairly well, but all the other coatings were unsatisfactory.—MR. 17232

5.3.2, 4.4.7

Protection Afforded by Metallized Zinc and Aluminum Coatings in Liquid Fuels. (In French.) Zbigniew Kowalski. Corrosion et Anticorrosion, **6**, 457-462 (1958) Dec.

Samples of steel coated with zinc or aluminum were immersed in five types of motor fuels for periods of 1128, 1248 and 2376 hrs. Presence of water and of tetraethyl lead affect corrosion resistance of these coatings, especially in case of zinc, but protection provided is adequate for walls of tanks and other vessels containing liquid fuels. 11 references.—RML. 17444

5.3.4

High-Temperature Carbonitriding of Plain Carbon Steels. (In Russian.) O. K. Kotov. Metalloved, i Obrabotka Metallov., No. 5, 16-20 (1958) May.

Discussion of experimental carbonitriding procedure—case compositions and depths obtained as function of holding temperature and time. S/N graphs are given for carbonitrided as against carburized specimens of 0.20 and 0.30 carbon steel and a 4 nickel, 2 chromium steel. Effects of various tempering temperatures on tensile, bend and fatigue characteristics are described. Comparison is given of mechanical properties obtainable by high-temperature carbonitriding as against carbonitriding at conventional temperatures, gas carburizing, high-frequency surface hardening and normalizing. Graphs.—INCO. 17443

5.3.4

Nickel Plating and Its Industrial Applications. (In French.) H. C. Castell. Paper before Conference Organized by Fabrimétal, Société Royale Belge des Ingénieurs et des Industriels and Centre Belge d'Etudes de la Corrosion, Brussels, May, 1956. Métaux, **33**, 366-376 (1958) Sept.

Review of current nickel plating processes, discussing bath compositions, plating procedures and properties of deposits, in sections on: Watts-type baths (effect of variations in pH, current density, temperature and chloride content), pure-chloride, sulfate, bright-nickel, bright nickel-cobalt, organic, sulfamate, tin-nickel, and nickel-phosphorus (electroless) baths.—INCO. 17224

5.3.4

Production of Zinc Coatings from Zinc Phosphate Electrolytes. (In Russian.) A. V. Rykova and E. I. Rudoy. Metalloved. i Obrabotka Metallov, No. 8, 33-37 (1957).

Steel cathodes were electroplated with zinc, using lead anodes, at 20-22C. Three acid baths were used. 1) zinc oxide 30-50, phosphoric acid 100-200 g/l; 2) zinc oxide 30-50, phosphoric acid 100-200, aluminum sulfate 20-25 g/l; 3) zinc oxide 45, phosphoric acid 115, nickel sulfate 110, total acidity 155 g/l. The deposit thickness

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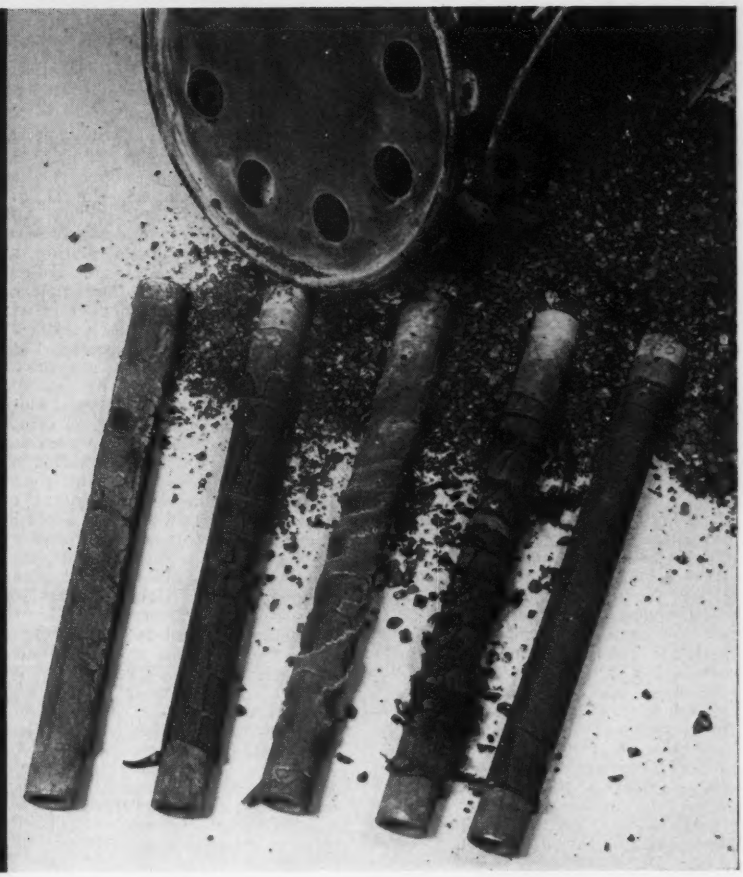

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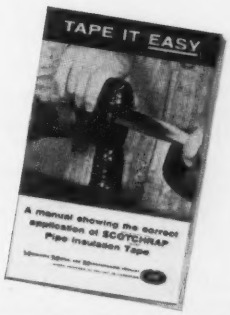

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was measured by a drop test and the porosity of the deposits by anodic treatment in a solution containing potassium ferrocy-anide and $Na_2SO_4.10H_2O$. The microhardness was also measured. From the results, given in detail, baths 1) at 6-10 amp/dm² and 2) at 10-15 amp/dm² are recommended for dense, non-porous deposits with good adhesion. Tests were also made with an alkaline bath. The deposits from all baths were easily polished to a mirror finish. The deposits could be chromium plated. The corrosion-resistance of the coatings was compared with that of nickel/copper/nickel coatings and chromium electrodeposits. During a 6-months' test in a humidity chamber, the zinc surfaces darkened after 2-3 days, but showed no significant change in weight.—MA. 17065

5.3.4

Bronze Plating from Modified Stannate Cyanide Baths. W. T. Lee. Trans. Inst. Metal Finishing, **36,** Pt. 2, 51-57 (1958/-59) Winter.

Account is given of variation in composition of bronze deposits with solution composition (metallic content, free cyanide and free caustic soda) and operating variables (current density, temperature). Solution changes (formation of sodium carbonate and sodium stannite) and correct balance of anode and cathode efficiencies are considered. Advantages of bronze plating compared with nickel and copper plating are reviewed. Throwing power of bronze solution is superior to that of sulfate nickel, cyanide copper and acid copper-plating solutions. This is particularly important since bronze deposits more than 0.0003 in. are not porous, and in this respect equivalent to 0.0008-0.001 in. nickel. Use of potassium-based bronze solutions gives improved electrode efficiencies. Satisfactory results have been obtained from pilot-plant operation of lithium-based solution. Table, graphs, photomicrographs.—INCO. 17449

5.3.4

Continuous Galvanizing by the Armco-Sendzimir Process. I. C. Gwynne. Sheet Metal Inds., **35,** No. 375, 497-506, 534 (1958) July.

Description of new line installed at Ebbw Vale Works of Richard Thomas and Baldwins Ltd. Preparation of surface and application of coating are discussed. Advantages of Sendzimir process over ordinary galvanizing processes are summarized. Photographs.—INCO. 17340

5.3.4

Cathode Potentials During the Electrodeposition of Molybdenum Alloys from Aqueous Solutions. D. W. Ernst and M. L. Holt. Univ. Wisconsin. Paper before Electrochem. Soc., Washington, D. C., May 12-16, 1957. J. Electrochem. Soc., **105,** No. 11, 686-692 (1958) Nov.

Cathode potential measurements were used to explain cathode reactions that result in electrodeposition of molybdenum alloys. Potentials were measured by direct method during electrolysis of aqueous ammoniacal citrate solutions containing sodium molybdate and sulfate of codepositing metal, iron, nickel or cobalt. Reduction of molybdate ion in this type of bath is accomplished with 1, 2 or 3 electrons depending on codepositing metal and its oxidation state in bath. Hydrogen is involved in molybdate reduction process. A 2-step mechanism for reduction of molybdate ion in presence of codepositing metal is proposed and explanation of why this metal must be iron, cobalt or nickel rather than other metals is presented. Tables, graphs. —INCO. 17241

5.3.4

Electrodeposition of Nickel Alloys from Pyrophosphate Bath. S. K. Panikkar and T. L. Rama Char. J. Electrochem. Soc., Japan, **25,** No. 11, 573-575 (1957) Nov.

Pyrophosphate complex salt is recommendable for the nickel plating, and is well known as a practicable reagent for copper plating. The negative polarization curves of nickel and copper in a pyrophosphate bath cross each other at one point, and this fact suggests the possibility of electrodeposition of nickel-copper alloy from the bath. The experiment was conducted to obtain successfully electrodeposition of various nickel alloys. For nickel-chromium, the bath contains nickel chloride, cupric pyrophosphate, potassium pyrophosphate, etc. The nickel content of the deposit varies 1-78 per cent according to the concentration of nickel and copper in the bath. For nickel-cobalt, the bath contains cobalt chloride and potassium pyrophosphate. Nickel content of the deposit is 40-92 per cent. In nickel-manganese deposition, manganese makes complex salt with pyrophosphate, and can never go above 0.05M in the bath. Nickel content of the deposit is equal to or above 91 per cent. For nickel-tungsten, the electrolyte concentration comprises nickel 0.3M (as chloride) and sodium tungstate 0.1M. The cobalt-precipitate has an appearance as of nickel-plated substance when the tungsten concentration is below 13 per cent. Burning may take place when the current density will exceed 2 A/dm². Further experiments are intended for the nickel-zinc alloy deposition.—INCO. 17073

5.3.4, 2.3.7

Cleaning and Preparation of Metals Prior to Electroplating. Effect of Oxide Films. Pt. XI. Effect of Oxides on the Adhesion of Electroplates. AES Research Project No. 12. H. B. Linford and A. Venkateswarlu. Columbia Univ. Plating, **45,** No. 7, 728-733 (1958) July.

Using techniques developed by Linford and Feder in their study of nickel plating on oxidizing copper, study was made of effect of oxide film thickness on adhesion of nickel plate. Modified Jacquet method was used to measure adhesion. Instead of plating for 12 seconds, as done previously, plating time of approximately 1 hour was used. Original oxide film thicknesses up to 1000 A did not affect adhesion of nickel on copper. As original oxide film thickness was increased up to 2500 A, adhesion decreased in regular fashion. When oxide thicknesses exceed 4000 A, nickel plate peels spontaneously during drying. Tables, graphs.—INCO. 17127

5.3.4, 3.2.2

Pitting in Thin Chromium Deposits. D. R. Millage and W. E. Hague. Udylite Research Corp. Tech. Proc. Am. Electroplaters' Soc., **45,** 118-122 (1958).

Type of defect on thick chromium deposits that is described as "low catalyst pit" and type that is actually a pore in chromium plate that is due to basis metal defect are described. Difference in results obtained when micro-hole is drilled in basis metal and thick chromium is deposited from hexavalent chromium plating solutions, with and without surface-active agent, are compared. Drilled micro-hole 2-4 mils in diameter will cause pore in 5-10 mil chromium plate deposited from chromium plating solution containing fluorinated surface-active agent (Zero-Mist). If surface-active element is absent, micro-hole is closed by thick chromium deposit. In case of thin deposits of chromium on nickel plate, presence of fluorinated surface-active agent in hexavalent chromium plating bath has no effect on intrinsic porosity of chromium plate. In thicker deposits there is beneficial effect in that stress-cracking is slightly reduced. Photomicrographs.—INCO. 17493

5.3.4, 3.5.9, 6.3.6

Thermal Shock Resistant Nickel Plating on Copper. L. Missel. Lockheed Missile System Division. Metal Finishing, **56,** No. 9, 49-51 (1958) September.

At exceeding high velocities, heat generated by air friction causes rapid deterioration of surface of copper parts. Use of this metal was functionally of great value and an investigation was therefore undertaken to provide protection for copper under conditions of thermal shock. Solar furnace tests were performed on a large number of metallic and ceramic coatings. Ceramics were all inherently porous, resulting in blistering and lifting of coating. Of all materials checked, nickel showed most promise, however, conventional plating techniques did not produce deposits which would withstand solar furnace shock. Nickel plated from a low stress sulfamate bath is less sensitive to thermal shock than nickel plated from Watts or other common baths. When taken above melting point of copper, alloying of nickel plated by conventional procedure with copper base takes place. Cupro-nickel alloys are well known to have extremely desirable physical characteristics. There is no evidence of failure of adhesion under these conditions. Tests indicate that nickel has a reflectivity considerably greater than that of copper at elevated temperatures. Photos, photomicrographs. —INCO. 17489

5.3.4, 3.7.4

The Influence of Physical Metallurgy and Mechanical Processing of the Basis Metal on Electroplating. Pt. IV. The Effect of Different Ferrous Metals on the Performance of a Watts Nickel Deposit. M. H. Jones, Chih-Yeu Lu and J. Zajdowski. Proc. Amer. Electroplaters' Soc., **44,** 53-69; disc., 162 (1957).

A statistical study of corrosion of a 0.0005- and a 0.001-in. deposit of nickel on to a selection of different ferrous metals shows that. 1) there is a significant difference in performance between certain metals for the two thicknesses; 2) the differences are smaller at the larger thickness; 3) there is a basic corrosion-rate which is not a function of the basis-metal composition but of the nickel deposit. The cause of the variation in the thinner deposit is related to comparatively large inclusions and materials having high-inclusion contents have poor corrosion-resistance. Non-conducting inclusions of silicon dioxide and aluminum oxide are an important source of deposit failure when the size of the inclusions is of the same order as the deposit thickness. These inclusions are not attacked by preplating procedures. Inclusions of semi-conducting materials, e.g. sulfides, may act as a source of pits when the deposit is thin, but their effect diminishes with increasing plate thick-

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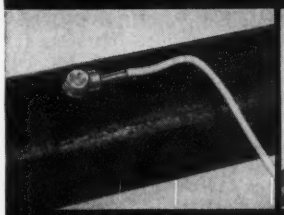

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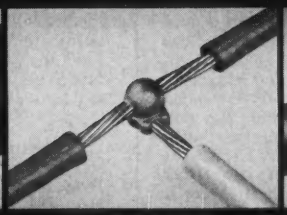

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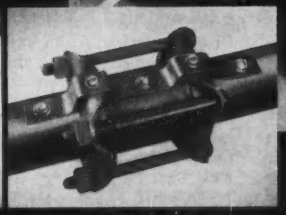

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ness. These inclusions are removed by a sulfuric acid dip or anodic treatment. The conditions under which inclusions are potential pores are not affected by the nature of the corroding agent. 14 references.—MA. 17013

5.3.4, 3.7.4, 3.4.7

The Influence of pH Value of Electrolytic Solution Upon Structure of Electrodeposited Nickel Film. (In Japanese.) Y. Wachi. J. Applied Phys., Japan, **26,** No. 4, 147-152 (1957) April.

A nickel film deposited from an electrolytic solution containing a suitable organic reagent such as 1-5 naphthalene disulfonic acid shows a characteristic structure. This phenomenon is assumed to be due to the increased cathodic potential originated by an absorption of organic reagent on the surface and also the deposit of nickel hydroxide resulting from the rise of pH value at the surface of the cathode. If the assumption is correct, pH value in a bulk solution may affect the structure of the deposited film. Observations on specimens deposited from a solution of pH value 1.5-5.5 are as follows: a) The surface of nickel film deposited from a solution of pH value 1.8 is similar to that deposited from an unadulterated solution. b) The preferred orientation changes suddenly at pH value 3-4, there appearing (200) orientation at the lower value and (220) orientation at the higher value in pH. The former orientation is of the same type as the film deposited in an unadulterated solution. c) A deposit at a pH value lower than 3 shows no "band structure" having no brightness, while a film formed at a pH value higher than 3 begins gradually to show "band structure." d) There is a tendency that the content of impurity of the deposit increases with the rise of pH value in a solution.—INCO. 17069

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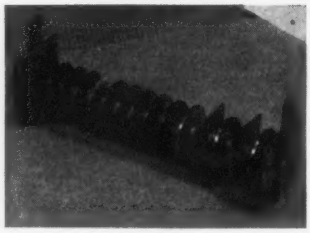

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6. MATERIALS OF CONSTRUCTION

6.2 Ferrous Metals and Alloys

6.2.1, 4.7

Static Corrosion Behavior of Construction Materials in an Environment of Liquid Bismuth Base Metals at 550 C. M. A. Cordovi. Brookhaven National Lab. U. S. Atomic Energy Commission Pubn., BNL-179, March 1, 1952 (Declassified Feb. 18, 1957), 40 pp. Available from Office of Technical Services, Washington, D. C.

This report presents preliminary results obtained in a static corrosion test program which was designed to screen promising construction materials for a proposed liquid fuel power breeder reactor. Iron, carbon steel, a graded series of ferritic chromium-molybdenum steels, and several stainless steels have been exposed for periods of 500 to 2000 hr to the action of bismuth, bismuth-lead eutectic and bismuth-lead-tin eutectic, with and without uranium (0.4% to 2% uranium) additions. All the experiments were conducted at 550 C, but one series of tests in uranium-bismuth was also carried out at 650 C. A novel type of corrosion sample was used in this investigation to determine quantitatively the extent of both surface and subsurface attack. The results obtained to date have no design significance and serve only to indicate general trends. Statistically proper evaluation of the results is impossible because the majority of the data were derived from single tests. Furthermore, the influence on corrosion rate of important incidental factors, internal or external to the metal, has not been given serious consideration in this preliminary phase of the corrosion program. The apparent trends exhibited by the various materials tested are summarized. On the basis of present information, it would seem that the steel and liquid metal combinations which show the greatest promise of success under the required temperature conditions are the 2¼ chromium-1 molybdenum and 5 chromium-½ molybdenum-1.5 silicon steels in uranium-bismuth and uranium-bismuth-lead (eutectic). (auth). —NSA. 15775

6.2.2, 5.4.2

Defects in Cast Iron for Enamelling Purposes. P. Gobbels. Paper before German Enameller's Assoc. Foundry Trade J., **103,** No. 2137, 363-366 (1957) Sept. 26.

Outlines some defects in cast iron that influence quality of final coating. Influence of wall thickness, causes of spalling of enamel coatings, effect of molding sands and casting conditions, and porosity defects are discussed.—INCO. 16196

6.2.1, 3.7.4

High-Performance Steels. E. A. Loria. Crucible Steel Co. of America. Machine Design, **30,** No. 18, 111-115 (1958) Sept. 4.

Discusses metallurgical fundamentals of microstructure, hardenability, and alloy composition as factors governing properties and, therefore, selection of steels. Effects of pearlite, bainite and martensite microstructures on steel properties are reviewed. Use of hardenability data is illustrated by Jominy test hardness versus section thickness data for 4340 steel. Effects of carbon content are emphasized. Avoiding of temper embrittlement by proper tempering is mentioned. Among graphs are those relating tensile strength to hardness for 4340 teel, and transition curves for bainitic micro-structures, and quenched and tempered 4340 steels.—INCO. 17477

6.2.2

Constitution, Production and Mechanical Properties of Ni-Resist. British Cast Iron Research Assoc. Research Report No. 488. R. Barton. J. Research and Development, British Cast Iron Research Assoc., **7,** No. 7, 299-315 (1958) Aug.

Survey of constitution, composition, structure, physical and mechanical properties of Ni-Resist. Range of total carbon, silicon, manganese, phosphorus, nickel, copper, chromium, tin, lead and arsenic in Ni-Resist is discussed. Austenite-to-martensite transformation is described. Thermal properties are tabulated. Data are given for following me-

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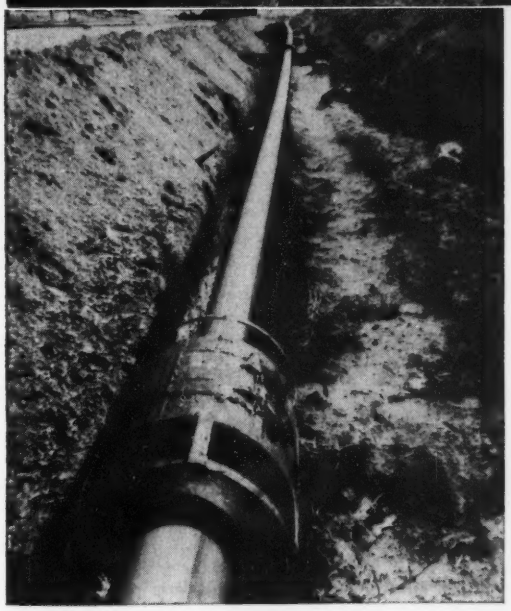

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chanical properties: transverse strength, compressive strength, torsional strength, elastic modulus, tensile strength, hardness, impact value, elevated temperature properties, creep and fatigue strength. Thermal growth resistance is considered. Cupola practice and foundry techniques are dealt with. Machining speeds are listed. Corrosion resistance to mineral acids, organic acids, alkalies, salt solutions and natural and industrial waters is reviewed. Brief list of typical applications is included. Photomicrographs, tables.—INCO. 17213

6.2.2, 3.7.3

Heat Treated Ductile Iron Offers Wide Property Range. C. A. Anderson. Baker Perkins, Inc. Iron Age, **182,** No. 21, 102-104 (1958) Nov. 20.

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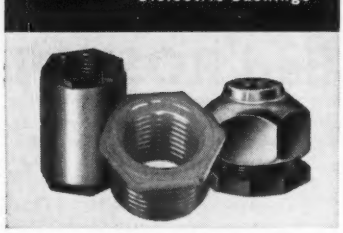

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Ductile iron comes in number of standard grades which, through proper heat treatment, can be modified practically at will. Besides 4 regular grades of ductile iron, there are 2 specials—a heat resistant grade, and ductile Ni-Resist. Chart shows how, with proper heat treatment (annealing, normalizing, oil quenching or flame hardening), it is possible to vary hardness, wear and shock resistance.—INCO. 17310

6.2.2, 8.8.5, 3.2.2

Formation of Undercooled Graphite in Cast Iron. I. Igarashi, G. Ohira, K. Ikawa and T. Horigomo. Trans. Am. Foundrymen's Assoc., **66,** 561-568 (1958).

Investigation to determine whether undercooled graphite solidified directly from melt or decomposed from ledeburite. It was found that both processes may occur according to circumstances. Effect of carbon, silicon, manganese, chromium, tungsten, molybdenum, oxygen, sulfur, selenium, tellurium and bismuth on graphite formation was studied and formation of undercooled graphite from liquid metal was discussed. Decomposition of ledeburite to graphite was investigated, with particular reference to effect of silicon. Graphs, photomicrographs.—INCO. 17333

6.2.2, 8.8.5, 3.7.3, 3.5.9

Study on Spheroidal Graphite Cast Iron. Pt. I. Heat-Treatment of Spheroidal Graphite Cast Iron and Its Mechanical Properties at Room and High Temperatures. (In Japanese.) H. Hotta and T. Saruwatari. Paper before Japan Foundrymen's Soc., Nov., 1956. J. Japan Foundrymen's Soc., **30,** No. 3, 137-144 (1958) March.

After having finished a preliminary experiment by melting the spheroidal graphite cast iron in the Tammann-furnace, a heavy oil furnace was employed to prepare specimens. By way of comparison, an auxiliary experiment was conducted with specimens made of ordinary cast iron, phosphorus rich cast iron and cast steel. In the hardness tests at room temperature and 400 C, an oil quenched spheroidal graphite cast iron showed a maximum hardness value which was being decreased in the order of the following heat treatments "quenched and tempered," "as cast," "annealed and slow cooled in the furnace." The difference in hardness of these samples at 600 C was very small. The hardness of ordinary cast iron was a little inferior to that of spheroidal graphite cast iron as cast, and it is almost equal to that of cast iron containing phosphorus. Hardness of cast steel is approximately the same as of annealed spheroidal graphite cast iron. In the wear resistance test between room temperature and 500 C, the tendency was the same as in the hardness test. In the impact resistance test at room temperature, spheroidal graphite cast iron showed a maximum value when it was annealed in a furnace. And its value was decreased in the order of the following conditions: quenched and tempered, as-forged, oil-quenched. In microscopic examination the structure of a matrix of oil quenched spheroidal graphite cast iron was martensite and that of tempered one was troostitic martensite.—INCO. 17342

6.2.3, 3.4.6, 4.6.2, 8.4.5

Carbon Steel in High Temperature Water. G. E. Moore. Oak Ridge National Lab. U. S. Atomic Energy Commission Pubn., CF-57-1-26, Jan. 31, 1957, 34 pp. Available from Office of Technical Services, Washington, D. C.

Resistance of carbon steel to corrosion in oxygenated high-temperature (250 C) water was unexpectedly good at high oxygen concentration. Pertinent literature, critically examined, and toroid experiments indicated that at low oxygen concentration attack did increase with concentration, but as oxygen concentration was sufficiently increased, more protective films were formed on the metal. Some corrosion factors in the application of carbon steel to nuclear reactor systems are discussed. (auth)—NSA. 16983

6.2.5, 3.7.2

Chromium, Nickel, Manganese Stainless Steels. J. R. Miller. Allegheny Ludlum Steel Corp. Western Machinery and Steel World, **49,** No. 10, 69-71 (1958) Oct.

From analysis of data obtained on corrosion, it was established that chromium content should be at least 17 per cent and increase toward 20 per cent depending on severity of application. Nickel content is helpful in more severe conditions, but can be eliminated in weaker environments. Manganese appears to have no influence on corrosion but is necessary to maintain austenite stability. Manganese content will be between 7 and 17 per cent depending on amount of nickel replaced. Nitrogen is normally limited to 0.25 per cent maximum for acceptable melting and hot working practices. Table shows specific compositions of family of alloys developed. While conservation of nickel was achieved, a new series of alloys was also developed which bridges gap in cost and performance between the straight chromium and chromium-nickel stainless steels. Compared with the 18-8's somewhat better mechanical strength, satisfactory corrosion resistance, equal fabricability and improved metallic brightness are all achieved at lower cost. Photos, graphs.—INCO. 17497

6.2.5, 3.7.2

Manganese Joins Aluminum to Give Strong Stainless. J. L. Ham and R. E. Cairns, Jr. National Research Corp. Product Eng., **29,** No. 52, 50-51 (1958) Dec. 22.

Alloying of aluminum and iron to give lightweight, stainless steel proved too brittle for cold working. Now Materials Branch of Navy's Bureau of Aeronautics is introducing a completely austenitic aluminum-manganese-carbon steel that supplies ductility for forming, and strength at high temperatures. First data are presented. Alloy contains 10.2 aluminum, 34.4 manganese and 0.76 carbon. It is 15 per cent lighter than carbon steel and 18-8, and 20-25 per cent lighter than nickel- and cobalt-base super-alloys. High strength is maintained throughout useful range of room temperature to above 1200 F. Oxidation resistance is adequate up to 1500 F. Tables compare mechanical properties of aluminum-manganese steel with carbon steel, Type 304 and Type 347.—INCO. 17347

6.2.5, 3.7.4, 3.5.9

Deformation Structures of an 18-10 Stainless Steel. (In French.) B. Jaoul. Rev. Met., **55,** No. 10, 976-992 (1958) October.

A few anomalies are pointed out by study of tensile curves of austenitic stainless steels. Various techniques (x-rays, micrography, mechanical properties)

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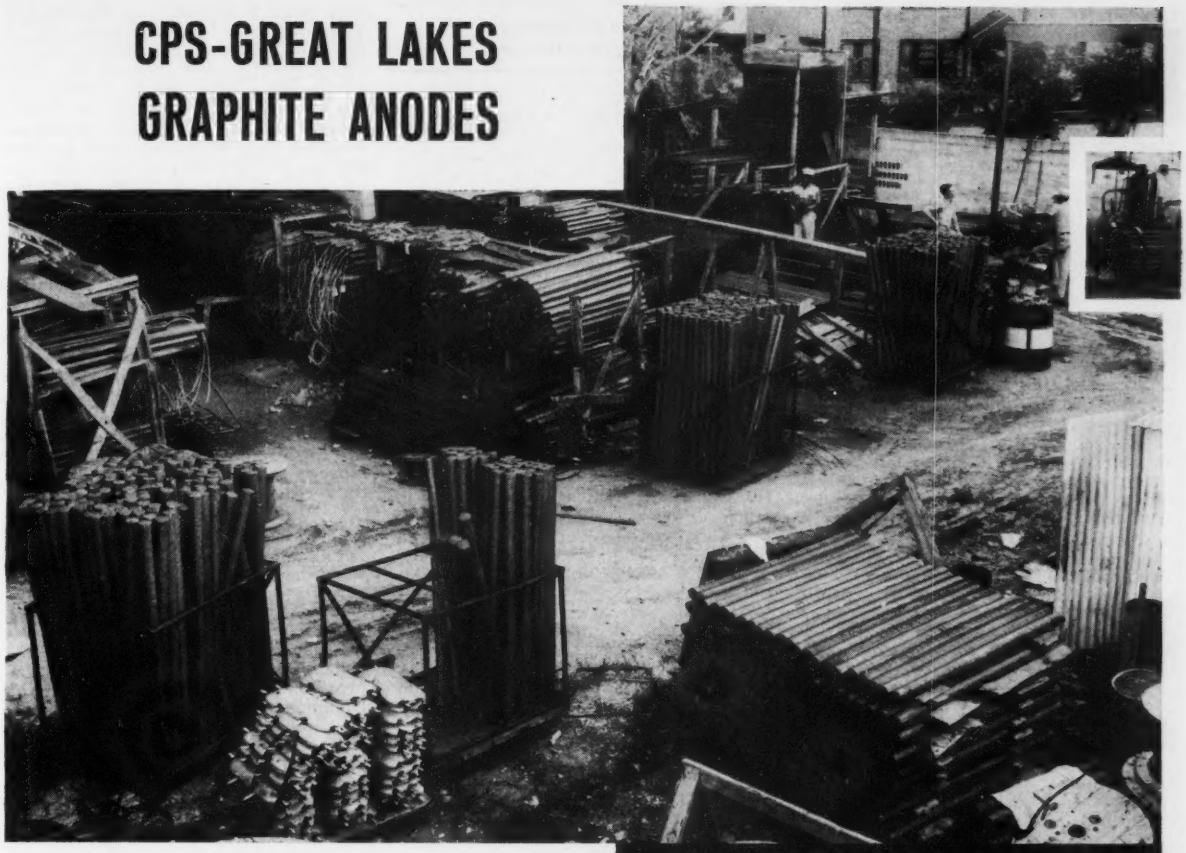

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make it possible to distinguish 2 kinds of deformation: below room temperature, a martensitic structure, the proportion of which depends on stress applied; and above room temperature, possibility of carbon diffusion brings about transformation of bainitic type. Graphs, photomicrographs.—INCO. 17381

6.2.5, 3.7.2, 4.3.2, 8.5.3, 3.2.2

Rust-Proof and Acid Resistant Steels (in Finnish with English summary). V. P. Ahonen. Paperi ja Puu (Paper and Timber), **40,** 97-102 (1958) March.

Stainless steels have found use in all sulfite pulp mill digesters built in Finland during the last few years. Chromium is the most important alloying element for preventing corrosion. Lowering the carbon content to 0.03 per cent makes steels immune to crystal-boundry or intergranular corrosion. In practice, however, a carbon content of 0.07 per cent is permissible. The surest means of preventing crystal-boundry corrosion is to alloy stainless steels with titanium, tantalum, or niobium. For resistance to pitting and to organic acids, stainless steels must be alloyed with 4 to 5 per cent molybdenum. The ratio of chromium/nickel/molybdenum is important. A steel with a 25:25:2 ratio of these alloying elements showed high resistance to corrosion under both active and passive conditions. Usually, 2 to 3 per cent copper also is added to increase resistance to sulfuric acid. A typical alloy steel containing chromium, nickel, molybdenum, and copper in a ratio of 18:18:2:2 was practically unattacked by pure sulfuric acid at 75 C in all concentrations and by boiling sulfuric acid of nearly 70 per cent concentration.—PDA. 17165

6.3 Non-Ferrous Metals and Alloys—Heavy

6.3.15, 3.7.2, 8.9.1

Titanium Alloys for Aircraft and Spacecraft. W. L. Finlay. Crucible Steel Co. of America. Western Machinery and Steel World, **49,** No. 7, 63-67 (1958) July.

Newer titanium sheet alloys are Formageable (formable in soft condition and ageable to high strength) alpha-beta DOD alloys—C 120AV, C 115AMoV and C 105VA—and Formageable beta alloy B 120VCA. Tensile and compressive strengths are discussed. Crippling strength of titanium alloys, aluminum and magnesium alloys, PH steels and Inconel X is considered; graphs show strength-density ratios at low stresses and at maximum practical stress. Aluminum exerts a controlling influence on long-term hot strength properties of DOD alloys—the higher the aluminum content, the higher the creep strength. In addition, the higher the aluminum content, the poorer the formability. C 120AV is in regular production while the other 2 DOD (Department of Defense) alloys are in pilot production and are being extensively evaluated in several airframe plants. B 120VCA is presently in pilot production and appreciable quantities are being evaluated by a number of airframe and missile manufacturers. Advantages of B 120VCA are listed. Graphs, tables.—INCO. 17269

6.3.15, 4.3.2

The Electrochemical Properties and Corrosion Behaviour of Titanium in Sulfuric Acid Solutions. (In Russian.) V. V. Andreeva and V. I. Kazarin. Inst. of Physical Chemistry, Academy of Sciences, USSR. Doklady Akad. Nauk SSSR, **121,** 873-876 (1958) August 11.

The corrosion resistance of titanium in solutions of sulfuric acid was investigated. Diagrams were plotted showing the rate of corrosion, potential and critical anode density of current in various sulfuric acid solutions.—NSA. 17174

6.3.15, 4.7

Corrosion of Titanium in Molten Salt Baths. (In German.) M. E. Straumanis and Y. P. Huang. Metall, **11,** No. 12, 1029-1032 (1957) Dec.

Corrosion of titanium in fused salt baths: new type of corrosion of metallic titanium in molten sodium chloride in presence of heavy metal salts; metal in salt precipitates on titanium specimens and provokes serious corrosion; only five out of 20 investigated salts, namely those of iron, nickel, copper, cobalt and cadmium were effective.—BL. 17043

6.3.15, 5.11, 4.3.2, 4.2.5

Titanium—Its Properties and Design Potentials. H. E. Barkan. Elec. Mfg., **62,** No. 4, 100-109, 272, 274, 276, 278 (1958) Oct.

Current structural and electronic uses for titanium and its alloys are presented. Potential uses based on exceptional properties in design of components and equipment are explored. Physical properties of standard and new alloys with fabrication data supported by charts are discussed. Graphs compare properties of titanium alloys with aluminum alloy. Type 347 stainless steel, S-816 alloy and 17-7 PH stainless steel. In corrosion

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resistance to mineral acids, titanium is somewhat similar to stainless steels. Titanium possesses excellent resistance to marine environments and passivates readily in a number of environments, exhibiting a noble electrode potential somewhat similar to that of a passive 18-8 stainless steel. Table.—INCO. 17183

6.3.15, 6.3.20, 8.8.5

Cast Titanium and Zirconium Parts. Stephen M. Shelton. Precision Metal Molding, **15,** No. 11, 48-49, 110-112 (1957).

The advantages of titanium and zirconium, and of casting methods rather than other processes of fabrication, are set out. The transparency to slow neutrons of zirconium, the use of titanium in aircraft and missile production and the anticorrosive properties of both metals are noted. Tolerances are given and typical parts illustrated.—MA. 17046

6.3.15, 8.8.5

Pilot Production, Fabrication and Evaluation of Promising Titanium Alloys. F. A. Crossley, B. R. Rajals and D. W. Levinson. Illinois Institute of Technology for U. S. Wright Air Development Center, U. S. Air Force, March, 1958, 46 pp. Available from Office of Technical Services, U. S. Dept. of Commerce, Washington 25, D. C. (Order PB 131978).

This report concerns the fabrication and evaluation of approximately 300 lbs. of titanium-7 aluminum-3 molybdenum alloy which was distributed to various jet engine manufacturers. One manufacturer reports success in producing 140 J-57 7th stage compressor blades from 70 lbs. of hot-rolled, centerless-ground bar. The endurance limit for these blades bench-tested at room temperature under completely reversed stress was 75,000 psi, 5000 psi higher than titanium-6 aluminum-4 vanadium alloy blades of the same configuration. The manufacturer reported that there were no difficulties encountered in processing the alloy and that it was not susceptible to oxygen contamination during processing. The tensile properties of the finished blade were also reported as superior. Another manufacturer produced wire in sizes down to 0.02-in. diameter from the alloy in $\frac{3}{32}$-in. diameter rods. Methods used in the preparation of the alloy are described. Results of other evaluations are included.—OTS. 17207

6.4 Non-Ferrous Metals and Alloys—Light

6.4.2, 3.7.4

New Properties and Uses of High Purity Metals. (In French.) Georges Chaudron. Rev. Met., **55,** No. 5, 407-416 (1958) May.

Properties of super purity aluminum prepared by the zone melting process, are reviewed. Results of studies carried out on the electric conductivity and recrystallization of aluminum at very low temperatures, as well as on competing effects, such as recrystallization and polygonization, and also on the intergranular corrosion of zone refined aluminum, are reported. It is pointed out that physical and structural phenomena in super purity metal usually develop in a relatively simple form which facilitates the study and understanding of them.—ALL. 16806

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6.4.2, 4.6.7, 4.6.4, 3.2.2, 3.4.8

Corrosion of Aluminium and Its Alloys by Waters of Various Compositions. (In German.) Otakar Sverepa. Werkstoffe u. Korrosion, **9**, No. 8/9, 533-536 (1958) Aug.-Sept.

The corrosion of aluminum and aluminum alloys by river waters and cooling waters of circulating systems was tested under natural conditions. While in river waters the corrosion attack was uniform and of low rate, in circulating waters it developed in the form of pitting. The composition of the water and the presence of impurities therein are said to have a predominant effect on the corrosion process. The differences between the corrosion behavior of various materials tested were found to be slight. It was established experimentally that aluminum-magnesium and aluminum-magnesium alloys suffer the most intensive corrosion by the combined action of Cu^{++}, Cl^{-}, Ca^{++} and HCO_3^{-} in presence of oxygen. Pitting corrosion depends largely on the concentration of the ions mentioned. Other cations and anions cause less pitting or none at all. Weight loss, the number of corrosion pits on the surface, the maximal depth of penetration and the nature of corrosion products as a function of Cu^{++}, Cl^{-} and Ca $(HCO_3)_2$ concentration, and also of pH value, were studied.—ALL. 16855

6.4.2, 3.7.2

Effect of Different Impurities on Corrosion Resistance of Super-Pure Aluminum in Water at High Temperature and High Pressure. (In German.) A. Domony. Acta Tech. Acad. Sci. Hung., **21**, No. 1-2, 123-139 (1958).

Corrosion resistance of high-purity aluminum alloyed with different amounts of silicon, iron, titanium and boron. Only aluminum containing certain kinds and amounts of foreign phase constituents was resistant. Corrosion behavior strongly enforced not only by formation of oxide films and electro-chemical behavior of phases against base metal but also by depolarizing effect of alloy phases. 10 references.—MR. 16964

6.4.2, 3.7.4, 3.5.4, 8.8.5

New Preparation and Properties of High Purity Aluminium. (In French.) Frédéric Montariol. Min. de l'Air PST 344, Booklet, 70 pp., 1958. Available from: SDIT, 2 Av. Porte-d'Issy, Paris 15°, France.

Experimental investigation of the preparation and properties of high purity aluminum. The specimens were irradiated in an atomic pile and used for the preparation of autoradiographs of the metal structure. It is shown that impurities are segregated in the interdendritic spaces of the structure; recrystallization tests indicate that the impurities are left in the final liquid stage, thus facilitating purification of the metal by zone melting. A purity of 99.999 percent can be attained. Such properties as ease of recrystallization and corrosion resistance are emphasized. 35 references.—ALL. 17486

6.4.2, 4.4.3

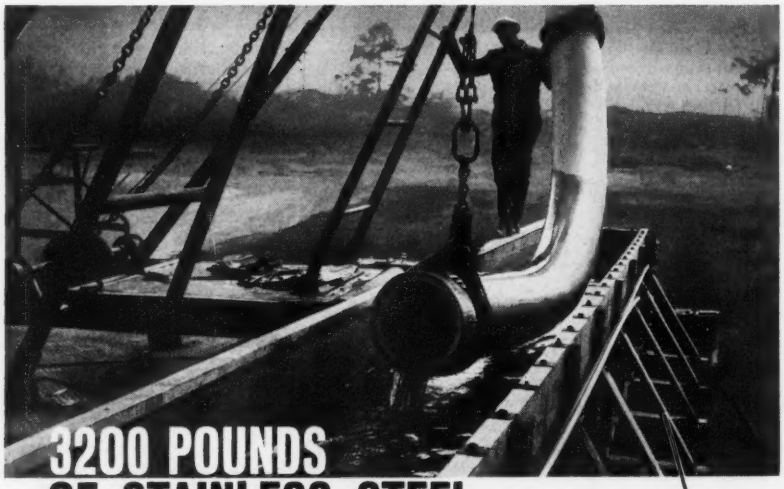
Investigation of the Behavior of Aluminum in Contact With Common Organic Solvents. (In German.) K. Broockmann. Fette, Seifen, Anstrichmittel, **59**, No. 12, 1078-1084 (1957) Dec.

Strong aggressive behavior of anhydrous, boiling n-alcohols and glycol monoalkyl ether against aluminum. Methyl glycol attacks pure aluminum at 60 C and very pure aluminum is attacked at 40 C. Control by water or ketone additions.—BTR. 17040

6.4.2, 4.6.2, 3.7.2, 3.5.9, 8.4.5

Corrosion of Aluminum Under Conditions Encountered in Nuclear Reactors. András Domony. Kohászati Lapok, **90**, 542-550 (1957); Chem. Absts., **52**, No. 21, 18149 (1958) Nov. 10.

Pure aluminum and alloys containing various amounts of iron, silicon and/or titanium were subjected to 1-6 cycles consisting of 8 hr exposure to steam at 180 C and cooling for 16 hr. The exposed samples were examined for tensile, microscopical, X-ray and chemical properties. Aluminum containing small amounts of silicon and titanium will corrode in relation to the temperature of the aqueous phase; at 180 C corrosion (I) begins after a few hr incubation, at >260 C within a few minutes, >0.2 percent iron will substantially inhibit I at 180 C (>0.3 percent at 260 C); >0.4 silicon, if no iron is present, will prevent I: 0.2 percent titanium will be effective at 180 C but not at 260 C. Boron will accelerate I unless silicon is also present, in the latter case it shows no appreciable effect. I proceeds inwards along the crystal boundaries and is attributed to the water (which is dissociated at high temperatures and pressures) in that the hydrogen ions will peptize the oxide layer at the surface which, in the absence of air, is unable to be regenerated. The depolarizing effects of the various ingredient metals, depending upon the potential differential, are of major effect in inhibiting or accelerating I.—ALL. 17032



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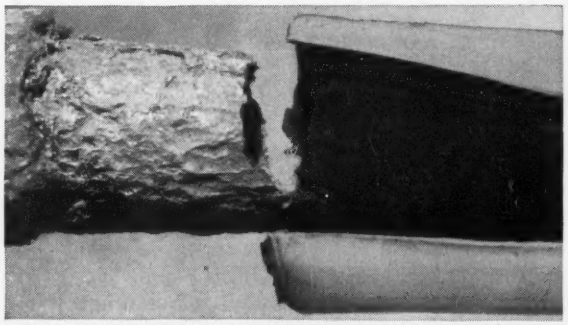

HOW X-TRU-COAT PREVENTS UNDERFILM MIGRATION. A section of coating was removed from this sample before it was subjected to an eight-month salt crock test. Corrosion ravaged the unprotected metal, but was unable to penetrate X-TRU-COAT's tight plastic-to-steel bond. Cutting back of the coating revealed *no underfilm migration!*

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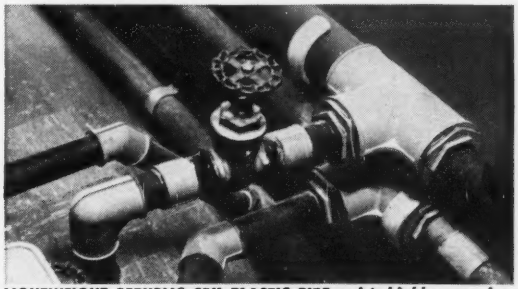

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7. EQUIPMENT

7.1 Engines, Bearings and Turbines

7.1

Where and How to Use Plain Spherical Bearings. A. R. McCloskey. Heim Co. Machine Design, **30,** No. 10, 119-122 (1958) May 15.

Plain spherical bearings greatly simplify design problems requiring high-capacity, self-aligning bearings and rod ends, particularly in mechanical linkages. Selection of bearing material is determined by conditions of load, speed, vibration, temperature and contamination. Ideal combination of materials at normal temperature, medium load, where loading may be either static or dynamic, is a finely finished through-hardened ball in a bronze bearing. Where temperatures exceed 500 F, stainless steel becomes necessary for both ball and outer member. Chromium plating and/or dry film lubricant are often essential to successful operation. Over 800 and up to 1200 F with or without corrosive atmosphere, balls of Stellite No. 3 and chromium carbide have been used in combination with Stellite or Inconel X for outer member. For chemical processing equipment and food-handling machinery where frequent cleaning is required, completely stainless bearings are customarily used. Photos, diagrams. —INCO. 16555

7.1

Operation and Constructional Evolution of Large Steam Turbines. P. Hummel. Brown Boveri Rev., **45,** No. 7/8, 310-324 (1958) July/August.

Describes excellent performance of new designs of reheat steam turbines, with special attention to steam circuits, starting process and certain constructional components. New developments are mentioned and standardization of low-pressure cylinder discussed. In a 150-MW unit (turbine fed with live steam at 171-180 Kg/cm² and 525-535 C with reheat to 525, max. 535 C), first stationary blades were designed as nozzles. Consequent absence of tip-leakage losses enables large heat drop to be used in this stage and reaction in first row of moving blades to be reduced without efficiency. Maximum temperature to which rotor is exposed is reduced, allowing low-alloy steels to be employed at still higher reheat temperatures. For 175-MW turbine under construction (live steam at 171-180 Kg/cm², 565-570 C with reheat to 540, max. 545 C), impulse blading had to be manufactured from 12 chromium steel to avoid scaling. Since there is as yet no known method for welding such steel to low-alloy steel containing chromium, molybdenum and vanadium, blades had to be mechanically fixed. Research is at present being carried out in attempt to solve this welding problem.—INCO. 16925

7.1, 3.7.3, 6.2.5

Welding and Stainless Maintain Water Turbines. L. McWilliams. Welding Engr., **43,** No. 7, 38-40 (958) July.

In maintenance of large water turbines, only economical solution for reclaiming worn guide-bearing sleeves is by welding stainless steel shells to them. Sleeves are first tack welded and then stainless steel shells are welded to mild steel sleeves using Type 301 electrode. Stainless steel has also been used to rebuild worn bronze sleeves; ECuSN-C type electrode is used. Pitted runners are reclaimed using mild low-hydrogen steel to fill cavities and applying protective overlay of stainless from Types 301 and 308 electrodes. Welding stainless steel overlay on worn areas is also used to repair wicket gate stems. Techniques are described.—INCO. 16909

7.1, 6.6.8

New Dry Bearing. R. E. Harmon. Machine Design, **30,** No. 15, 22-24 (1958) July 24.

A new bearing composition, called DU, has just arrived from Great Britain—a composition that has some radical properties as a dry bearing material. Chief characteristics of the new material are its excellent bearing properties when run dry, its high strength and its good thermal capabilities. Compared to other dry bearing materials, the new composition seems to be outstanding. Its ancestor, DP-1 was a tetrafluoroethylene-plastic impregnated, sintered porous spherical bronze overlay on a steel backing strip. Marriage of 20% metallic powdered lead with 80% TFE fluorocarbon plastic has made essentially a new bearing material which has properties far surpassing the earlier compositions. Properties are described.—INCO. 16869

7.1, 8.9.1

A Study of the Metallurgical Properties that are Necessary for Satisfactory Bearing Performance and the Development of Improved Bearing Alloys for Service Up to 1000 F. G. K. Bhat and A. E. Nehrenberg. Crucible Steel Co. of America for U. S. Wright Air Development Center, U. S. Air Force, November, 1957, 73 pp. Available from Office of Technical Services, U. S. Dept. of Commerce, Washington 25, D. C. (Order PB 131609).

Numerous efforts by the aircraft industry to use hot work die steels and other tool steels for bearings have been hampered by a lack of information on the properties of the materials at high temperatures. This research was aimed at obtaining such material properties as hot hardness, compressive yield strength, resistance to softening and structural and dimensional stability. Twenty-nine steels were examined, ranging in type from SAE 52100 and its modifications, to hot work and other tool steels. Although none of the steels studied appeared suitable for application at 1000 F, the following were found satisfactory for elevated temperature aircraft bearing application: Halmo 1 (room temperature to 700 F); VSM, M50, M10 and an experimental grade (Room temperature to 800 F); and T1, M2, M1, and another experimental grade (room temperature to 900 F). In addition to possessing optimum metallurgical properties required in high-speed bearings, the steels also are comparatively lean in alloying elements.—OTS. 15925

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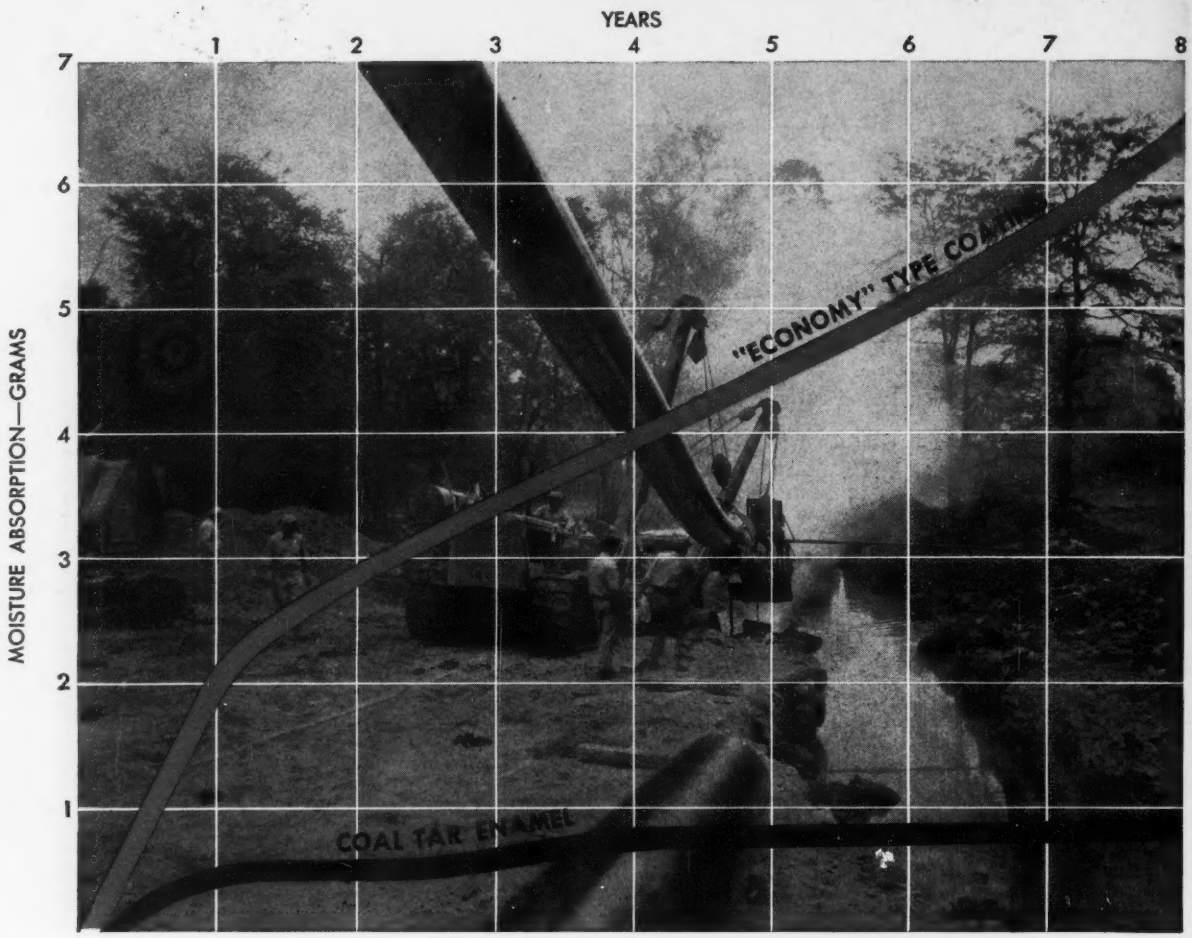

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